

METALLURGIA

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(INCORPORATING THE METALLURGICAL ENGINEER)

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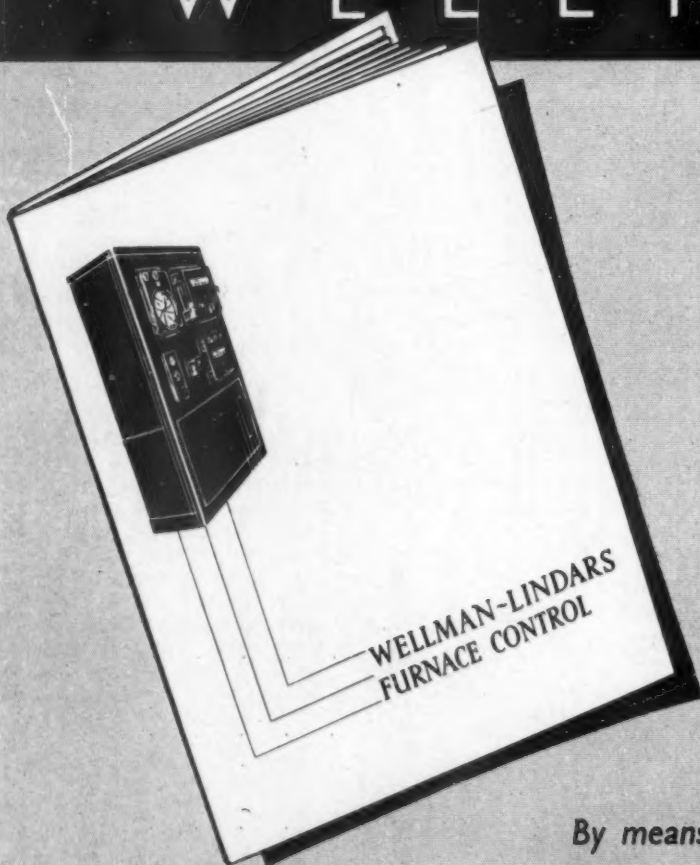
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METALLURGIA

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British Steel Supplies for 1947

ALTHOUGH the present decade has seen an amazing expansion in the production and use of non-ferrous metals, particularly the light alloys of aluminium and magnesium, the ferrous materials still account for something of the order of 90% of the output of all metals. The products of the iron and steel industry are so important as to be vital to civilisation, indeed, no other material has contributed so much to the welfare and comfort of mankind. Yet the importance of iron and steel is frequently overlooked, due probably to the fact that supplies have generally been available to meet demands. During the war, of course, the industry was called upon to produce as near as possible to plant capacity, and since demands for the Forces increased, control became necessary to ensure the proper direction of the iron and steel available. Although the change from war to peace caused some dislocation it was comparatively slight and the position remains in which the demand for the products of the industry exceeds supply. Thus, reorganisation, to which the industry is committed, must be carried out while endeavouring to meet the pressing demands for more and more iron and steel.

Of the products of the iron and steel industry, steel is of primary importance, although developments are in progress to increase the production of the basis iron. Production of steel in 1946 amounted to 12,693,000 ingot tons, compared with 11,824,000 ingot tons in 1945. During the past year output was affected to the extent of nearly 500,000 ingot tons because of shortage of coal. It is noteworthy that the highest output of steel recorded in this country in any peace-year was 12,984,000 ingot tons in 1937.

The question naturally arises: What are the prospects for this year? It has been authoritatively stated that the output of steel during 1947 should be between 13 and 13½ million ingot tons; that under favourable circumstances 13½ million ingot tons could be produced. To achieve this adequate supplies of fuel, transport and scrap will be necessary. Although imports are extremely difficult to forecast, it is expected that 750,000 ingot tons will be received from Dominion and other sources. This is about the minimum pre-war import. On the question of export, the Government will permit only steel exports declared essential, thus it is almost impossible to forecast the tonnage of steel to be used for this purpose, but it is unlikely that more than a million ingot tons will go abroad, indeed, it is probable that the amount will be less than a million tons. As approximately 250,000 ingot tons of steel are still due to come from stocks of war steel held by the Government, the total supply of steel available for home consumption during 1947 will be between 13 and 13½ million ingot tons.

In suitable areas open-hearth furnaces are being rapidly converted to oil firing. One ton of oil is equivalent to about two tons of coal, and thus augments our fuel supplies in the present shortage. Furnace capacity using 400,000 tons of oil will have been converted by the end of the first quarter this year, and the industry expects to be using oil at the rate of a million tons annually before the end of the year. Whether this conversion to oil fuel can be regarded as an emergency measure or not, it is difficult to say, but it does indicate the precarious position of the coal industry upon which British industry has been built. It is true, of course, that some oil is obtained in this country, but the major amount consumed is imported, and when the coal industry has been thoroughly reorganised and output substantially increased, it is likely that economical considerations will result in a return to the use of coal.

A feature of the supply position was the marked rise in home demand for steel during the latter half of 1946. During the twelve months June, 1945, to June, 1946, home consumption was running at the rate of approximately 11 million ingot tons. During the last six months of 1946 home consumption rose rapidly to a rate of 11½ million ingot tons. In order to strengthen the nation's exchange position, and at the Government's request, the industry, in the early part of 1946, exported a considerable tonnage of steel. Last March, however, it was recognised that a heavy home demand for steel was bound to materialise over the following nine months and continue thereafter. In consequence a drastic reduction of exports was made and as much steel as possible diverted to home industries.

This diversion of steel to home consumers has enabled many industries, such as the shipbuilding and motor-car industries, to achieve very high levels of production. On shipbuilding, it will be remembered that Mr. Barnes, Minister of Transport, announced last month that, in the third quarter of 1946, ninety-three vessels were launched in Britain, of a gross tonnage of 268,713 tons. This can be regarded as a remarkable performance, especially when coupled with a further statement by Mr. Barnes that Britain had under construction a greater tonnage than the rest of the world put together. The motor-car industry has been allocated a tonnage of steel for the first quarter of 1947 and if this allocation can be maintained throughout the year it will suffice for a production of 400,000 cars for the year. And it should be remembered that the best pre-war figure was 389,633 in 1937. It is hoped, however, that the allocation of steel to this industry will be increased later in the year.

The possibility that the iron and steel industry may be able to provide 13 to 13½ million ingot tons of steel for home activities during 1947 indicates that this favourable position in these vital industries may even be improved. Consider the production figures during

the four years 1936 to 1939 inclusive which were: 10,765,000, 11,621,000, 9,238,000 and 12,811,000, respectively. It will be noted that the prospective tonnage exceeds that of 1939, when war programmes dominated. The average tonnage of the four years mentioned—11,108,750 ingot tons—indicates the improved prospects for the consuming industries this year. Indeed, it is confidently believed that the over-all flow of steel to consuming industries during 1947 will not be far out of line with their ability to use it. Indications of this are given by the employment figures, which show that in 1938 the total employed in industry was 17,100,000; the corresponding figure in September, 1946, was 17,800,000 excluding Armed Forces. The proportion of workers in the metal-using industries is higher than before the war, but is now showing a tendency to decline.

The severe limitations imposed by the shortage of fuel, and transport difficulties, must also restrict manufacture of many products involving the use of steel, such as motor cars, ships, railway rolling stock, housing and domestic requirements, as well as numerous other products. Many shortages, like timber and glass, cannot be easily overcome, but steel is being used as a substitute for many vital raw materials.

In considering the prospective production of steel for this year the broad view should be taken that if more steel is available, either from a better raw material position at home, or from a more normal flow of imports, it should be possible to increase the direct export of steel above the figure given and thus reduce the sacrifice of overseas goodwill, at present being made in favour of home consuming industries. In view of the Government's allocation system it is impossible to give a close approximation of the distribution; if available steel is possible the steel industry will certainly do its utmost to make the tonnage available as large as possible, but Government Departments decide who is to have steel and how much.

From the long-term point of view, part of the present demand for steel must be regarded as temporary, but as the various consuming industries develop, a permanent increase in home demand of steel would seem to be certain and the steel industry is now constructing new capacity which will give facilities for a further two million ingot tons per annum. As is well known, modernisation schemes in various works throughout the industry were held up when war commenced as it was considered more economical of man-power to obtain extra steel from America. Now the industry has much leeway to make and will need to build as quickly as possible the additional capacity needed without interfering with production from existing plant. For this purpose a five-year plan is in operation. It is confidently stated that when the modernisation programme is completed, Britain will have a highly efficient iron and steel industry.

The plan is the result of a combined effort by the whole industry and its leading technicians, many of whom, with the full authority of their companies, made great sacrifices to ensure as near possible perfection in the finished modernisation proposals. Actually an Efficiency Committee was set up in May, 1944, to prepare the framework of the post-war plan. The work of this Committee was based on development schemes already approved by the British Iron and Steel Federation before the war. The greater part of this Committee's proposal was published in outline in July, 1945, before the General Election results were known, and as will be appreciated,

there was a natural hesitation on the part of the companies concerned to go ahead with their modernisation proposals, which interrupted progress during the early part of last year. Fortunately, the setting up of the Steel Board gave the companies greater confidence and enabled the industry to resume work on the plans and, in addition to the completion of considerable preparatory work, a large number of smaller projects are now either nearly completed or, in some cases, being run in.

A brief reference to these smaller projects will be of interest. They include a new rod mill at Richard Johnson & Nephew, Ltd.; the new cold reduction reversing strip mill at John Summers & Sons, Ltd.; new blast furnaces at Consett Iron Co. Ltd.; modifications to the tube plant at Stewarts & Lloyds, Ltd.; iron foundry fitting shop at Whitehead Iron & Steel Co. Ltd.; electrolytic tinning line at Richard Thomas & Baldwin, Ltd.; new steel melting shop at the Appleby-Frodingham works of United Steel Companies, Ltd.; blast-furnace and steel furnace developments at the Scunthorpe works of John Lysaght, Ltd.; new ore unloading plant at Dorman, Long & Co. Ltd.; blast furnaces at Clyde Ironworks and at Guest, Keen, Baldwins, Ltd. These smaller schemes in the general plan will undoubtedly increase the over-all efficiency of the industry. In addition much greater attention is being given to research with a view to improving the working of plants as well as the products of the industry.

The prospects for the industry are good and as difficulties, at present encountered are overcome, output will increase as the year proceeds and will undoubtedly reach new high levels giving that impetus to consuming industries of benefit to the country as a whole.

Magnesium-Zirconium Alloys

ALTHOUGH considerable progress has been made in recent years in the technique of fabricating magnesium alloys, no major development has been associated with new alloys for several years. During the past twenty years or so, much work has been carried on with a view to the development of new alloys, but although many patents have resulted the older magnesium alloys have retained their positions of superiority. Research work during the past two or three years, however, has shown the value of zirconium as an alloying element and in this and the following issue the results of this extensive work is described by Major C. J. P. Ball.

It is noteworthy that experimental research in this field was undertaken in Germany in 1938-39, when it was found that zirconium appeared to exercise an intensive grain refining effect on magnesium, but the problems of effectively and economically introducing zirconium into magnesium alloys were not easily solved and the work was stopped. However, the research staff of Magnesium Elektron, Ltd., decided to continue with a view to the solution of the problems. Steady and persistent work has resulted in the production of magnesium-zirconium alloys possessing properties of an outstanding character. Not only have they exceptionally high proof stress which, combined with high ultimate stress and elongation, may revolutionise the light alloy casting field, but their relative ease in working opens great possibilities for the wrought alloys. The particular attention of readers is directed to the data which will be published in our February issue.

Outstanding Properties of the Magnesium-Zirconium Alloys

By Major C. J. P. Ball, D.S.O., M.C., F.R.Ae.S.

Industries to-day are searching for stronger and ever stronger structural materials, and in particular for metals of low density combined with the highest possible properties, to help, for instance, in improving the efficiencies of new turbine engines. In this paper, presented by the author at the First International Magnesium Congress recently held in New

York, the efforts of Magnesium Elektron, Ltd. towards the achievement of these objectives, are summarised and particular attention is directed to the value of zirconium as an alloying element. Steady and persistent work has led to the production of magnesium-zirconium alloys possessing properties of an outstanding character. In addition to their exceptionally high

proof stress which, combined with high ultimate stress and elongation, may well revolutionise the ultra light alloy casting field, their relative ease of working coupled with very high mechanical properties opens great possibilities for the wrought alloys. Data which will be given, in the concluding part, will indicate that progress of a high order has been achieved.

IT was the writer's good fortune through the past war to be permitted to take part in the phenomenal expansion of magnesium production in the United States, which assisted so materially the Allied Air Forces in the advancement of victory. The joint efforts of Britain and the United States raised the combined productive capacity of the two countries, within the short space of four years, from around 16,000,000 lbs. per annum to nearly 672,000,000 lbs. per annum, a technical achievement which well deserves to be placed on record as a great task well done.

The greater part of this expansion took place on the American side of the Atlantic, and the resolute yet audacious manner in which the United States tackled her construction and production problems astonished the world. There can be no doubt that the formation of the Magnesium Association to use the experience gained in war to create an ever-increasing consumption of magnesium products deserves the highest possible praise, and affords a shining example of what can be done rapidly to adapt a vital war potential to peace-time usage.

Just as steel and aluminium, and in fact, most metals, have constantly increased their field of usage by improving the quality of their alloys and lowering the cost of fabrication, so magnesium in turn must strive to follow the same road. The high cost of production of the wrought forms of magnesium, particularly sheet, have exerted a most adverse influence upon all attempts to increase usage; the need to reduce the cost of fabricating is,

therefore, one of our most pressing problems.

It is especially important that the magnesium industry should be able to offer alloys with materially improved properties and workability, because designers and engineers in the aircraft and other industries to-day are searching eagerly for stronger and ever stronger structural materials, and, in particular, for metals of low density combined with the highest possible mechanical properties, to help them to improve yet further the efficiencies of their new turbine engines, to lower the structural weight of their ever-growing aircraft, and generally to reduce weight and combine lightness with strength.

Figs. 1 and 2 which illustrate typical large castings in regular production for jet engines in England show how greatly existing magnesium alloys are

contributing to this marvellous new power plant, and it is certain that with improved mechanical properties they will become of even greater value to the engineer.

Some indication of the efforts and of the progress made by M.E.L. (Magnesium Elektron, Ltd.) during the recent difficult years towards the achievement of these objectives is given in the following brief summary. It will be understood that this progress has only been made possible by the untiring work of the Company's chemical and metallurgical staffs, operating as teams in the closest possible accord, the chemists solving the problems of preparing the required alloying constituents and making them directly from the ore, and the metallurgists then attacking the problems of alloying and preparing the best alloys.



By courtesy of the De Havilland Aircraft Co. Ltd.

Fig. 1.—Front casing for a De Havilland jet propulsion unit (Goblin I). Casting by Sterling Metals Ltd.



By courtesy of Rolls-Royce Ltd.

Fig. 2.—Rear blower casing for a Rolls-Royce jet unit. Casting by Sterling Metals Ltd.



Fig. 3.—A large zirconium-containing magnesium alloy ingot.

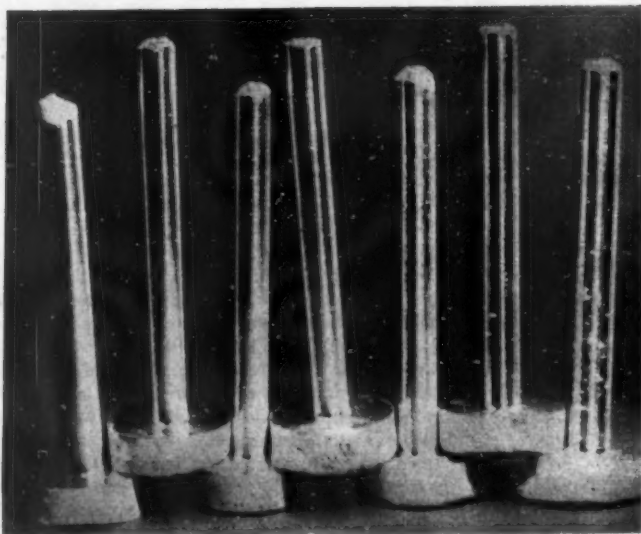


Fig. 4.—Z3Z alloy made by the initial chloride process, re-melted and cast into sand bars, machined and exposed to damp air to show up flux inclusions. (Note the widespread primary chloride contamination particularly in the last bar).

Summary of Progress

It is a most interesting commentary upon the magnitude of the problems involved that although magnesium alloys have been accepted as engineering materials for more than twenty years, yet, during the whole of that period, scarcely any new alloys of outstanding merit have been discovered and developed. It is certainly not for want of trying, as the patents in all countries, both post- and pre-war, clearly indicate; but, as few of the new ideas have proved successful in practice, the alloy systems in general use remain the magnesium-aluminum-zinc series, and the magnesium-manganese series, sometimes with third or fourth elements added.

Experimental research by I. G. Farbenindustrie in 1938 and 1939 disclosed to M.E.L. that zirconium used as an alloying element appeared to exercise a most intensive grain-refining effect on magnesium. For example, the grain size of ordinary pure magnesium when chill cast can vary from say 2 mm. to a much greater size, while the same metal containing an effective zirconium addition of 0.65%, in the case of the very massive chill casting shown in Fig. 3 having dimensions approximately 70 in. \times 20 in. \times 20 in., will have a grain size of 0.05 mm. to 0.15 mm. It was soon found that this grain refining effect can be further intensified by the addition of other alloying elements at the same time as zirconium. For example, the

grain size of an alloy containing about 0.65% zirconium and 4½% zinc in the chill cast form would be about 0.03 mm.

Although I.G. had made the initial discovery, they found that the problems of effectively and economically introducing zirconium into magnesium alloys were not easily to be solved, and early in 1939 they dropped all work on the subject and recommended M.E.L. to do the same. Experimental lots of I.G. zirconium alloys were brought to England by M.E.L. in 1938 and 1939, but the lack of effective fabricating technique prevented any successful usage by consumers.

Because the results of these early experiments appeared to support the belief that the physical and mechanical properties of magnesium alloys can be greatly improved by reduction in grain size, and disclosed that the zirconium alloys might provide, in both the wrought and cast states, considerably higher proof and ultimate stresses with good elongations, than were so far obtainable with the known magnesium-base alloys, M.E.L. decided to disregard I.G.'s advice, and to continue to try to solve this most interesting problem.

The minimum properties suggested for specifications are given later, but some idea of the figures which then awakened our interest is especially noteworthy. In the cast state, for instance, with a simple heat treatment, 0.1% proof stress figures of 10 tons/

sq. in., and in the wrought state, 0.1% proof stress figures of 20 tons/sq. in., can readily be obtained. Impact values, notch sensitivity, and fatigue characteristics are good, and the corrosion resistance is outstanding.

In a recent paper, Aitchison* said: "A high proof stress ratio is one of the dearest wishes of the aeronautical engineer," probably one of the most important statements made on materials for a long time. It is confidently believed that the zirconium alloys materially improve the claims of magnesium alloys in this respect.

As will be readily understood, the problems met with were extremely complicated, and were found to be as much chemical as metallurgical, necessitating a large number of physico-chemical and metallurgical investigations. On the chemical side it was necessary to determine the best and cheapest processes for making directly from the ore the most effective and suitable type of chloride alloying constituents, to enable the work to proceed.

On the metallurgical side the work was more straightforward, though by no means simple, owing to the complex metallography of the zirconium alloys, the need to introduce and maintain sufficient zirconium in the most effective form to ensure maximum grain refinement leading to good mechanical properties, the difficulty of

* Prof. L. Aitchison, paper read before the Royal Aeronautical Society, April, 1946.

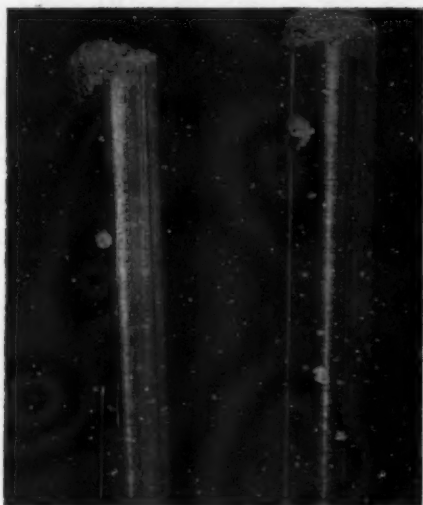


Fig. 5.—Second and last bar from the same melts in Fig. 4, showing flux contamination.

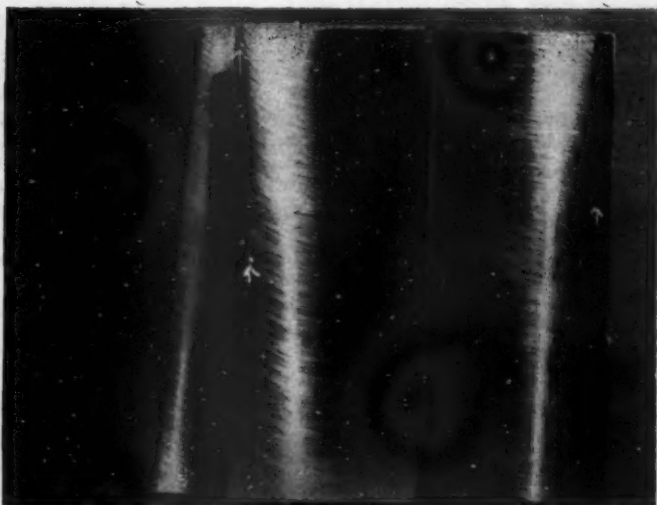


Fig. 6.—Flux contamination resulting from the use of inappropriate fluxes.

determining the content of effective zirconium by analytical methods, and the sensitivity of their mechanical properties to certain trace impurities.

By 1943, progress was so rapid that success seemed to be assured and draft specifications were submitted to the Ministry of Aircraft Production. However, before the new alloys were made available, or even experimentally used outside the laboratories of M.E.L., extensive tests of the alloy product were undertaken to check the corrosion resistance and general freedom from

inclusions. It was a disagreeable surprise to find from examination of two-ton melts that the metal contained small centres of chloride contamination disseminated in minute pinpoints through the metal. Examination of this chloride contamination by chemical and spectrographic methods disclosed it to be quite unconnected with the melting and refining fluxes used, and to consist of the chloride reaction product resulting from the alloying reaction, which, unlike all other types of flux inclusions, persisted on remelting.

Intensive efforts were made to remove these inclusions, or to prevent their formation by many and various methods, none of which was successful.

The examples shown in Figs. 4, 5 and 6 show how great a problem was encountered in this form of flux contamination. They show defects which vary from:—

(i) Widespread primary chloride contamination of the pin-point type indicated, to

(ii) Infrequent but more serious type of flux contamination which inevitably results if inappropriate fluxes are

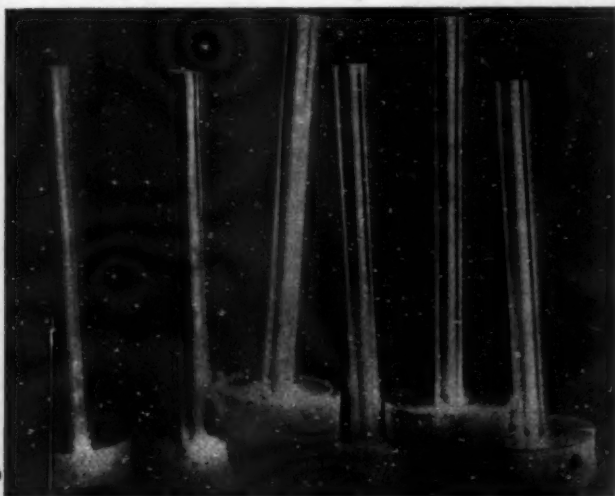


Fig. 7.—Elektron ZSZ sand-cast bars made by the fluoride process using special fluxes, machined and exposed to show possible flux inclusions. (Note the complete absence of flux inclusions even in the last bar).

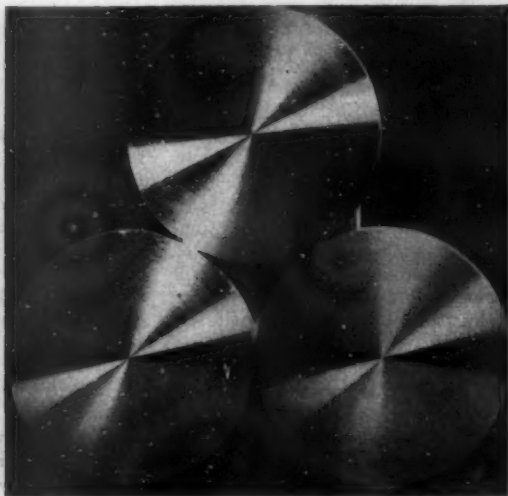


Fig. 8.—Slices from a 7-in. dia. ZBF billet made by the fluoride process. (Note the complete absence of flux contamination).

employed, quite irrespective of whether or not the alloy suffers from primary flux contamination.

Fig. 9.—Photomicrograph of Elektron A8 unsuperheated.

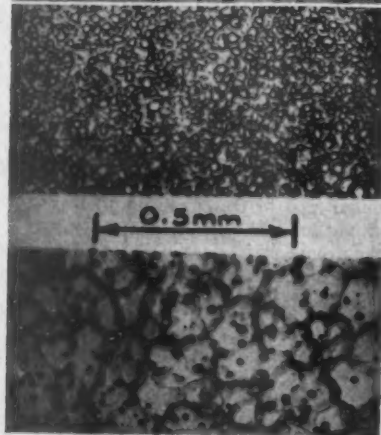
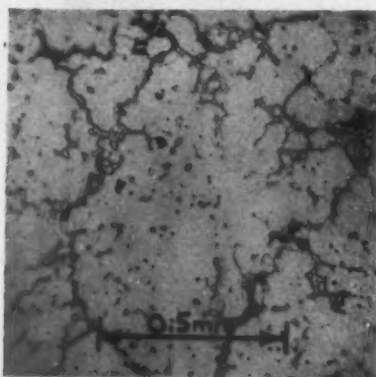


Fig. 10.—Comparison of the structure of A8 superheated with die-cast Elektron Z5Z.

Compare these latter illustrations with the clean metal prepared by our fluoride processes (shown in Figs. 7 and 8). It will be noted that the zirconium alloys as now made are completely free from flux inclusions. In addition, Fig. 9, which shows a normal standard alloy (Elektron A. 8) before grain refinement, and Fig. 10, showing the same after grain refinement, side by side with a zirconium alloy of grain size 0.05 mm., clearly illustrate the intense grain refinement induced by zirconium.

Parallel with this work on the chlorides, experiments were proceeding on a smaller scale using metallic zirconium as the alloying constituent. It was found that if the magnesium was heated to a very high temperature of the order of 900° C. to 1,000° C., preferably under argon, effective alloy-

ing was possible though difficult and much too costly.

Undismayed by this disappointing setback, and with their determination to achieve success stimulated by the excellent grain structure and mechanical properties so far obtained, M.E.L.'s staff sought new methods of introducing zirconium into magnesium. Exploratory work was started more or less simultaneously with the following alloying substances: zirconium metal, potassium fluozirconate, zirconium fluoride, zirconium oxide, zirconium nitride and zirconium sulphide. Of these only the fluorides showed any promise of success.

Unavoidable preliminaries of this work which has taken in all some two years to complete, were the extensive clarification of the chemistry of the fluorides of zirconium which have turned out to be a most complex group of bodies, and the development of processes and plant for the production of the more suitable fluorides and fluozirconates (i.e., combinations of zirconium fluoride with the fluorides of more electro-positive elements).

Study of the methods of preparation of zirconium fluoride with satisfactory alloying properties showed that the existing literature on this compound was very incomplete and in fact misleading. Much work was needed to establish which were the most suitable forms of the normal, the basic and the hydrated fluoride, how these might be prepared and their quality controlled. These substances can now be prepared at costs which are very much less than would be expected for compounds of a comparatively rare metal such as zirconium, and are indeed comparable with the cost of many common metallic fluorides.

As the experimental work progressed it was found that the greater reducibility by magnesium of fluorides when compared with chlorides led to undesirable impurities as well as zirconium entering the metal. By an exacting study of salt mixtures and equilibria these impurities were eliminated, and the cause of severe explosions resulting from the use of fluorides of inappropriate form and composition tracked down and avoided.

During the investigations it was found to be quite readily possible to make up an "alloy" which contained several per cent. of zirconium as revealed by analysis. However, the body so obtained was not a real alloy, since further work established that not more than about 0.75% to at most 0.8% of zirconium is soluble in

magnesium-base alloys. The manner in which the zirconium acts in reducing the grain size of the alloy is not yet clear, but it does seem to be plain that a large part of the zirconium addition enters into solution in the magnesium-base matrix (this solubility, both in the liquid and solid state, appears to be affected by temperature), while some of the zirconium may be present in a state of extremely fine division which may almost be described as colloidal. Attempts were made to differentiate by a chemical test between the soluble zirconium (i.e., the zirconium which is really dissolved in the solid matrix) and the insoluble zirconium by a chemical test, soluble zirconium passing into solution when dissolved in reagents of appropriate concentration, and the insoluble zirconium not passing into solution. Unfortunately the results did not prove reliable.

Physical methods for determining effective zirconium content were then examined and one selected which in practice proved to be most reliable, and which disclosed that the oxide and metallic zirconium alloying methods even at their best were unsatisfactory. With this information available fresh progress was made and the alloying process improved to permit the temperature of zirconium introduction to be lowered from around 900° C. to 780° C.

On applying the new process to zinc containing alloys, difficulty was at once found in introducing sufficient zirconium. This led to a study of factors influencing loss of effective zirconium from the molten alloys on which quantitative work had been impossible prior to the introduction of a physical method for determining the effective zirconium content. Some eight principal causes of zirconium loss were established, and methods found to avoid them, so that finally zirconium could be introduced into the magnesium-zinc alloys just as successfully as into pure magnesium; but the old problem of flux inclusions still persisted, although in a greatly reduced form. Many and various methods were tried to remove these inclusions and achieve perfectly clean metal, but of all these only that of settling under favourable temperature gradient effected any real improvement.

The adoption of the technique of settling under a favourable temperature gradient led to the preparation of special high density fluxes of the inspissated type for use with zirconium-containing alloys. Using these heavy fluxes, completely flux-free

zirconium alloys are obtained and satisfactory methods of preparing zirconium alloys for wrought purposes on a 2-ton scale are now in operation; slab and billet can be cast in large sizes (e.g., billets of 70 in. \times 20 in. \times 20 in.). Methods have also been worked out for remelting satisfactorily most types of residue metal.

Because the presence in the alloys of traces of the electro-positive elements, whose fluorides are used for alloying, has an adverse effect on sand-cast mechanical properties, new methods for preparing alloys intended for sand castings had to be worked out. Since it is more convenient for producers of sand castings to receive zirconium alloy in ingot form rather than to perform the whole alloying process themselves, a method had to be found to reduce the zirconium loss, which at present inevitably occurs on remelting the alloys, and to provide a means of revivifying remelted zirconium ingot so that the full properties are restored without introducing either chloride contamination or traces of undesirable elements.

This problem proved difficult of solution, but eventually a master alloy, and also a master salt, were developed which perform these functions effectively.

Later an alloy for high temperature work (named Elektron MCZ) was developed in co-operation with Messrs. J. Stone and Co., Ltd.; this contains 3% mischmetal as well as about 0.7% zirconium and has excellent properties, shown in Fig. 15.

Metallography

The purely metallographic side of zirconium alloys is of great interest. A number of insoluble zirconium-rich phases may be present and the elucidation of the composition of these has involved much work. Zirconium alloys show peculiar forms of coring and the relations between coring and grain boundaries are sometimes most curious and complex. When the studies are complete it is intended to publish an account of the work and the conclusions reached, but it will be of interest to examine Figs. 11-14 which show the grain and coring structure of an alloy containing 0.7% zirconium and 4.5% zinc, named Elektron Z5Z. (Zirconium rich in centre of core, zinc rich at grain boundary).

Corrosion

The corrosion resistance of zirconium alloys is much better than the best of the present normal purity alloys and compares favourably with

the corrosion resistance of the ultra high purity materials. It is thought that their excellent corrosion resistance may be due to the formation of a complex and resistant oxide film containing both zinc and zirconium, and that the low iron content also helps. The alloys also give promise of enhanced resistance to pitting.

Zirconium alloys can be protected satisfactorily by the standard treatments of the D.T.D. 911A specification. It has been found, however, that some of the treatment solutions, in particular the acid chromate bath,

have a short life when treating zirconium alloys. A new bath, the M.E.L. chrome manganese bath, was therefore devised specially for these alloys; the film produced has good protective properties and provides a satisfactory base for paint.

Generally, it may be said that the zirconium alloys have proved to have corrosion qualities better than the normal purity standard alloys and only slightly inferior to high purity standard alloys. They present no difficulties in protective treatments or in painting.

To be continued.

Fig. 11.



Fig. 13.

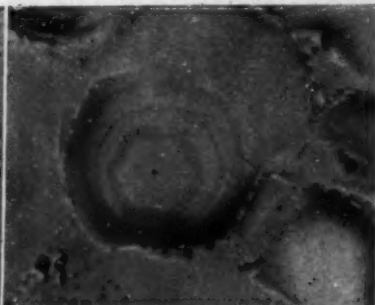


Fig. 12.



Fig. 14.



Figs. 11-14.—Grain and coring structure of Elektron Z5Z. (Zirconium rich in centre of core, zinc rich at grain boundary).

Atomic Hydrogen Welding

PROGRESS in the welding of light gauge materials used in the aircraft industry can be measured by the increased knowledge shown in welding design, making fuller use of existing welding facilities, and the introduction of special alloys used in the work. According to Dever* the method now in use with the atomic hydrogen welding process demand a slower welding speed than was used earlier, yet this is still a little more than twice as fast as the oxyacetylene method. The use of the "silent arc" has become standard because of the finer control that is obtained.

Higher hydrogen pressures are used in tacking, as well as a higher current since, as is common in most tacking operations, a high heat is required to bring the metal temperature up quickly to develop a small weld without distorting the assembly. The tungsten electrodes are adjusted so that the arc fan can be rotated to almost any desired angle.

Types of joints welded with this process include: lap, fillet, butt, flange, seam, edge and corner.

The major function of a welded joint is the final bond obtained after welding is complete, its soundness, strength and fatigue resistance characteristics.

* F. S. Dever, *Welding Journ.*, 25, 1946, 309-312.

Carbon and Alloy Steels for General Service Applications

Specially Contributed

Schedule B.S./S.T.A.5, revised and issued for the Ministry of Supply as a permanent document, for common use at all stages from development to production, is reviewed. Attention is directed to additions of detail, to additional steels included, and to the amplification of notes on their selection.

IN 1942 a schedule of service steels, B.S./STA5, for armament and vehicle manufacture was issued for the service departments by the British Standards Institution. This schedule¹ was drawn up by a conference of the technical representatives of various Government Departments in collaboration with the Superintendent, Technical Applications of Metals, Ministry of Supply, and was prepared as a statement of the general service requirements for engineering steels to co-ordinate and simplify the material aspect of war-time manufacture. It rationalised steels used by the services and laid down a basis for the unification of requirements by the various user departments. It also formed part of a complete scheme which embraces the non-ferrous materials, copper, nickel, lead, tin, zinc and aluminium, and their alloys² (magnesium has still to be dealt with); the spring steel specifications S.T.A.1, 2, 3 and 4³; and other more specialised requirements covered by separate specifications in the S.T.A. series, including one, S.T.A.25, for high silicon iron castings issued in May, 1945.

In December, 1945, Schedule B.S./S.T.A.5 was revised and issued for the Ministry of Supply by the British Standards Institution as a permanent document for common use by all concerned at all stages from development to production. As the previous data contained in it had in general been found satisfactory, few modifications were found to be necessary, but it was considered desirable in the light of experience to make considerable additions of detail. A few additional steels have also been included and the notes for guidance in their selection have been amplified. In view of the growth of welding fabrication, particular attention has been given to this aspect of usage and general notes on the subject have been included, as well as additions to the notes given for each individual steel. The section dealing with conditions of supply has been recast, and a slightly different arrangement of specifications has been made, with a view to greater clarity in use.

Free-Cutting and Carbon Steels

The new schedule contains two free-cutting steels, V1A and V1B, in place of the former V Steels, 1, 1A, 1B and 1C. The first steel is recommended for lightly stressed components not subject to shock (nuts, studs, etc.), suitable for production on automatic lathes; and the second steel, which contains higher manganese, is suitable for parts where good machinability and finish is important, but where the disadvantages of the higher sulphur required to give full free-cutting properties makes the use of the more rapid machining steel, V1A, undesirable.

¹ Reviewed in METALLURGIA, July, 1943.

² Reviewed in METALLURGIA, Sept., 1943; April, 1944; Aug. 1944; Dec. 1945.

³ Reviewed in METALLURGIA, April, 1944.

The former mild steel, V2, suitable for cold-working, is replaced by six mild steels, V2, V2A, V2A/1, V2B, V2C, and V2D, suitable for cold-forming as bars, billets, forgings, plate, sheet, strip, wire and tubes. The compositions of these steels and their minimum mechanical properties as bars, billets, and forgings are given in Table I. V2 is a general purpose cold-working steel for simple pressings, while V2A and V2A/1 are suitable for more difficult pressings, containers, etc., V2B is suitable for difficult pressings where greater strength is required and also for containers, wheel rims, frame members, rivets, etc., V2C is unsuitable for deep pressings and only simple forms can be cold-pressed economically in the heavier gauges, while V2D should only be specified when 28 tons ultimate tensile stress is an essential minimum, such as for chassis frames, brackets and cold-formed structural members.

TABLE I.—COMPOSITION AND MECHANICAL PROPERTIES OF V2 STEELS AS BARS, BILLETS AND FORGINGS.

Steel	Composition				Ult. Tensile Stress Tons per sq. in.	Elong. % 4 area minimum	Bend 180°	Brinell Hardness
	C.	Mn	S	P.				
V2 ..	0.20 Max.	0.80 Max.	0.06 Max.	0.06 Max.	30 Min.	28	R=FT	—
V2A ..	0.12 Max.	0.50 Max.	0.05 Max.	0.05 Max.	18/26	35	Flat	120 Max.
V2A/1	0.08 Max.	0.50 Max.	0.04 Max.	0.04 Max.	18/26	35	Flat	120 Max.
V2B ..	0.08 Max.	0.50 Max.	0.05 Max.	0.05 Max.	30/26	30	Flat	120 Max.
V2C ..	0.15 0.20	0.40 0.60	0.05 Max.	0.05 Max.	26/32	28	R=FT	—
V2D ..	0.17 0.25	0.40 0.70	0.05 Max.	0.05 Max.	—	—	—	—

As regards the other carbon steels V3 remains unaltered, V4 includes special composition requirements ranging from V4/1 to V4/4, V4A includes free machining quality V4AM and special composition requirements: V4A/1 to V4A/3, V5, include one special composition requirement, V5/1 and V5A two special composition requirements, V5A/1 and V5A/2, V5A and its modifications are steels containing 0.6-0.7% carbon and are new additions. For all those carbon steels, additional welding notes are given, and more applications for which each steel is suitable are also included.

Alloy Steels

To the three carbon manganese steels, V6, V6A and V7, a new carbon-manganese steel, V6B, of welding

quality and suitable for cold-forming has been added. This steel is specified to contain 0.16-0.21% carbon and 0.8-1.2% manganese. Its applications are similar to those of V6A, but where a more ductile and more readily weldable steel is required. The manganese-nickel-molybdenum steel, V8, suitable for welded structures requiring high tensile properties, remains unaltered, but the manganese-molybdenum (low alloy steel), V9A, and the manganese-molybdenum (higher molybdenum), V9B, are specified in special composition requirements, V9A/1 and V9B/1. In the former the carbon is reduced from 0.30-0.38% to 0.25-0.30%, and in the latter 0.30-0.50% of chromium is added. Four special compositions, V9C/1 to V9C/4, containing from 0.3-0.35% carbon to 0.27-0.32% are given, for the 1% chromium steel and two special compositions, V9D/1 and V9D/2, containing 0.35-0.4% and 0.40-0.45% carbon respectively for the 1% chromium-molybdenum steel.

The three nickel-chromium-molybdenum steels, V10, V11 and V12, which are used for highly stressed transmission components remain unchanged. Two 4½% nickel-chromium steels, V13 and V13A, suitable for air-hardening are specified for highly stressed gears and other transmission components where freedom from distortion in heat-treatment is required. Both steels contain 0.26-0.34% carbon, 3.9-4.3% nickel and 1.1-1.4% chromium. V13A contains in addition, 0.2-0.4% molybdenum. The 1% carbon-chromium steel, V14, for parts requiring a high hardness remains unaltered.

Case-Hardening and Nitriding Steels

In Table II are given the nine case-hardening steels included in the new schedule, three of which, V15A, V17 and V18B, are additional steels. With the exception of the 2% nickel-molybdenum steel, V16B, each of these steels has a lower carbon variety containing a maximum of 0.12% carbon and designated V15/1, V15A/1, etc. Steel V16B has two high-carbon composition requirements, V16B/1 and V16B/2 containing 0.2-0.252 and 0.23-0.28% of carbon respectively.

TABLE II.—CARBURISING STEELS AS BARS, BILLETS AND FORGINGS

Specification	Condition. Core properties 1½ in. dia. test sample	Ult. Tensile Stress Tons per sq. in.	0.2% Proof Stress Tons per sq. in.	Elong. % 4 area	Mod. ft./lbs.
V15	Carburised and Heat-treated	32	22	20	40
V15A	"	40	25	20	40
V15AM	"	40	25	20	30
V16	"	45	30	18	40
V16B	"	55	40	15	25
V17	"	45	26	18	40
V18	"	55/70	45	15	35
V18A	"	65/80	55	12	30
V19	"	85	65	12	25

V15 and V15A are general purpose case-hardening steels, the former suitable for applications requiring moderate core strength and the latter where slightly higher core strength is required. Steels V16A to V18A are used for gears, shafts, pinions and other transmission components, and V19 for high duty gears, heavy roller

bearings, etc. The case-hardening 2% nickel-molybdenum steel with higher carbon, V16B, has been successfully used in place of 5% nickel case-hardening steel.

For the nitriding 1½% chromium-aluminium-molybdenum steel, four compositions are now specified, V20A to V20D. These contain 0.2-0.25%, 0.25-0.35%, 0.35-0.45% and 0.45-0.55% carbon respectively, the other elements including 1.4-1.8% chromium, 0.10-0.25% molybdenum, and 0.9-1.3% aluminium being the same for all four steels. With all these steels a minimum diamond pyramid hardness of 1,000 may be specified, but where this is not essential and the core properties are important a minimum hardness of 800 D.P.H. may be specified. Two additional nitriding steels are given in the new schedule. These are chromium-molybdenum steels, V21A and V21B, containing 0.1-0.2 and 0.2-0.3% carbon respectively, 2.9-3.5% chromium and 0.4-0.7% molybdenum, and a minimum surface hardness of 800 D.P.H. may be specified for either steel. All the nitriding steels are suitable for parts highly resistant to wear, steels V21A and B being used where maximum hardness is not considered essential.

Spring Steels

Carbon spring steel for oil-hardening and tempering, V22, is specified in the new schedule in two compositions, containing 0.65-0.85% and 0.45-0.65% carbon respectively, as V22A and V22B. Closer ranges of carbon within the overall limits of the specification give four special composition requirements, V22A/1, V22A/2, V22B/1 and V22B/2. All these steels are suitable for hardened and tempered small springs, clips, washers, magazine springs, cartridge chargers, etc.

Laminated springs and torsion bars are specified in silicon-manganese steels V23 and V23A respectively. Both steels contain 0.55-0.65% carbon, 1.7-2.1% silicon and 0.8-1.0% manganese, but steel V23A must be of a very high standard of cleanliness and must be free from stringers, slag threads or aggregates of crystalline silicates or oxides as revealed by macro-etching. An inclusion count may be specified when required, and it is desirable that the phosphorus and sulphur contents shall not exceed 0.03% each.

Special Steels

For internal combustion engine valves two steels containing chromium and silicon are specified. The first, V24, contains 0.4-0.5% carbon, 3-3.75% silicon, 3-6% manganese and 7.5-9.5% chromium, and the second, V24A, contains 0.74-0.84% carbon, 1.75-2.25% silicon, 0.2-0.6% manganese, 1.15-1.65% nickel and 19.5-20.5% chromium. The latter steel is only used for valves operating under severe conditions.

Chromium rust-resisting steels in the new schedule are V25, V25A and V25B, including free machining qualities V25M, V25AM and V25BM and special composition requirements V25B/1 and V25B/2. These steels contain 12-14% chromium and have carbon contents ranging from 0.18-0.35% with the exception of the V25B steel in which the carbon is 0.18% maximum. In the free machining qualities sulphur may be present up to 0.75% maximum and selenium, zirconium, molybdenum to a 0.6% maximum, and lead to a 0.35% maximum may be added to a total of not more than 1%. All these steels are suitable for applica-

tions requiring corrosion resistance combined with some degree of mechanical strength, such as water pump spindles, roller tracks, etc.; V25B/1 is used for turbine blades.

High nickel-chromium-tungsten valve steel V26, for internal combustion engine valves working under special high duty, high temperature conditions remains unchanged. Rust and acid-resisting austenitic steels included in the schedule are the 18/8 type, V27, included in the former schedule with additional varieties including a welding quality, V27A and a free machining quality V27M. A new rust-resisting high tensile chromium-nickel steel of the 18/2 type is added and is designated V28. Steels V27 and V28 are specified where greater corrosion resistance is required than is obtained by V25.

Welding

In an appendix to the schedule general notes on welding are given and such notes together with those included in the individual specifications are intended only as a general guide, indicating the probable scope of the steels in respect of welding, outlining the procedure to be followed, and directing attention to the necessary precautions to be taken.

Steels which rely on cold-working (cold-rolled or cold-drawn) to provide the required mechanical properties, will have these properties altered locally by the heat of welding and in the majority of cases the strength is considerably reduced by welding. Steels which rely on heat-treatment to provide the required mechanical properties will also have their properties altered locally by the heat of welding. In both conditions consideration must therefore be given in design to the local alteration of properties in the vicinity of welds. If fusion welding is employed the weld may have properties that differ from the parent metal.

For a weldable steel, there is no maximum limit to the thickness which can be successfully arc-welded, but, from an operating point of view there is a minimum limit of thickness of $\frac{1}{16}$ in. for arc-welding, below which other welding processes are more suitable. The weldability of steel decreases with an increase in the percentage of carbon and other alloying elements it contains—the effect of carbon being the most important and far greater than that of other elements. As a low-carbon high-alloy steel will therefore weld more readily than a high-carbon low-alloy steel, the designer should choose a steel of a composition to give the best weldability for the mechanical properties required.

The zone adjacent to a weld, which is transformed by welding heat, may be hardened by the quenching effects of the neighbouring mass of cold steel, and the hardness of this hardened zone can be kept down by reducing the cooling rate of the weld and adjacent plate by increasing the diameter of the electrode and the cross-sectional area of each run of weld metal and also by preheating. Where preheating is recommended in the schedule, it is for the purpose of reducing thermal shock and not for stress-relieving and it is essential in these cases that the minimum preheat temperature recommended be maintained during the whole welding operation.

Post-heating in welding may take three forms:—

(a) Reheating the welded component to a definite temperature to reduce stresses set up from cooling of the weld;

(b) Steels which depend on heat-treatment for their physical properties may be welded prior to heat-treatment when the alteration of properties due to welding cannot be taken care of in design. In such cases the whole component after welding is given the treatment necessary to give the required properties and to relieve the weld stresses.

(c) Following the weld with a flame to retard the rate of cooling, thus reducing any tendency to harden due to cooling of the weld. The recommended temperatures for pre-heating and post-heating when necessary are given for each steel in the schedule. In certain cases, particularly with steels of high carbon and alloy content, it is necessary for post-heat treatment to be carried out immediately after welding.

Welding Alloy Steels

In welding alloy steels it is recommended that the largest electrode and the heaviest run that can be employed should be used, as it has been shown repeatedly that with the narrow runs deposited from small electrodes—particularly as first runs—the weld bead is very liable to crack away from the plate. Where multi-run welds are used, they should preferably be deposited in such a manner as to retain the heat in the plate, i.e., subsequent runs should be deposited as soon as possible after the previous run. Attention should also be paid to the details of tack welds in order to prevent dangerous hardness being developed.

It is also stated that austenitic electrodes of a suitable type, rather than mild steel or high tensile electrodes, will generally be found necessary when steels with a high alloy content have to be welded. As the ultimate tensile strength of the weld metal from such electrodes is 40–45 tons per sq. in., due account of this strength should be taken in the design of structures involving steels of higher strength. When welding steels high in carbon and alloy content, the practice of pre-coating joint surfaces with 25/20 or 18/8 electrodes prior to infilling with 18/8 weld metal is advantageous in avoiding cracking. Mild steel or high tensile steel should never be deposited over austenitic weld metal or the deposit will contain cracks.

Flame and Resistance Welding

In flame welding the same precautions apply in general as for metallic arc welding, but special preheating is often unnecessary as sufficient preheat over a large area is automatically provided by the flame itself. For satisfactory welding precise and consistent control of the flame is essential.

The principles which govern the need for preheating and post-heating in the case of metallic arc and flame welding apply also to resistance welding (seam, spot, projection, butt and flash-butt), as the quenched effect is even more severe. When using steels, with which this quenching effect may cause cracking or embrittlement, it is necessary to adapt the machine to post-heat and/or pre-heat. Unless this can be arranged, difficulties are likely to be encountered in spot, projection and seam welding, except in the case of mild steel containing less than 0.18% carbon and free from residual alloying elements. It is essential in all cases, that the steel be clean and free from oxide film.

The Chemical and Physico-Chemical Analysis of Iron and Steel

Five Years' Advancement

By. E. C. Pigott

In this article the author gives an exhaustive and descriptive bibliography of contributions on ferrous analysis published during the period 1942-46 inclusive. The progress achieved during the period 1930-41 inclusive was depicted by the author in a series of articles published in this Journal during 1942. The present contribution brings the bibliography up to date and will, we believe, be of value to many readers. It will be noted that developments in analytical methods continue to become more direct in order to meet demands for speed combined with accuracy.

Introduction

AN exhaustive and descriptive bibliography depicting the progress achieved in ferrous analysis during the period 1930-41 has already been contributed to this journal by the author*. It was accorded a reception so pleasing as to prompt the compiling of this present and further contribution bringing the bibliography up to date.

Attention was drawn to the prevalence not only of the mere rearrangement of minor details of procedure but also of actual repetition of investigations. Judging from such repetition, which continues to be in evidence, it is plain that the laudable practice of exploring the relevant literature before embarking on method research could be more widely followed. A regrettable handicap—the dearth of authoritative translations—has been largely remedied by the Iron and Steel Institute which, incidentally, has thereby compensated to some extent for the relative rarity (with the possible exception of gaseous constituents) of analytical contributions to its Journal.

A notable trend of the past five years has been the remarkably extensive development and adoption of spectrographic analysis, ordinarily inapplicable to the determination of carbon, phosphorus and sulphur, yet far more rapid than chemical procedure for most other constituents, especially where the number of analyses is considerable. Moreover, the samples are readily prepared and the record is a permanent one. From the qualitative standpoint, the spectrograph, if it were more portable, would have no serious rival. Quantitatively, the highest accuracy obtains where large numbers of casts are melted to a single specification, mixed batches entailing a loss of precision. So useful is the instrument for quickly determining the content of the accidental elements that this particular application alone would ensure a popular place in the well-equipped laboratory. On the debit side, the spectrograph can be applied only to the estimation of elements whose log-ratio curves are known, while the maximum determinable amount of a constituent is strictly limited. Again, the area excited is so minute that results are liable to be vitiated by heterogeneity. Nor is the technique readily applicable to drillings.

The outstanding difficulties which to-day stand in the way of further development are light-source instability and inadequate reproducibility of the spectral excitation.

Quite frequently the errors are far greater in magnitude than those in chemical procedure and, were it not for specification margins, might well outweigh the advantages of speed and economy. Of much importance is the successful application of the spectrograph to the analysis of segregates.

A striking feature of the period under review is the progress made in photoelectric absorptiometry. In this country, much of the development has centred around the very few proprietary types of instruments here available. A very interesting instrument which can be constructed from standard components employs a divided selenium barrier-layer cell on the grounds that two independent cells would undergo differential fatigue. Balancing is effected not by means of light apertures, but by means of a variable resistance network. The 40 mm. solution cell is fixed in position. In the United States more attention has been paid to the devising of highly selective colorimetric procedures, generally suitable for any of the numerous one-cell and two-cell instruments marketed there. Rectifier and photoemission photocells are generally employed, but photoconductive types are also used. While the tendency has been to equip the instruments with arbitrary scales a few are designed to give transmittency, extinction or concentration of constituent directly.

Chemical preparation of the coloured solution can be greatly curtailed where a photoelectric spectrophotometer is used, in view of the selectivity of the monochromatic beams. Certain American types of this instrument are equipped with automatic recording mechanism, permitting as many as 150 determinations daily. The operator must be sufficiently skilled to bring the components into adjustment, after which, however, he has but to place the test solution in position and to start the motor operating the wave length cam, the curve then being plotted automatically. Reliable microchemical methods of truly astonishing sensitivity owe much of their success to the photoelectric absorptiometer.

An instrument that has fully justified its early promise and has gained a firm foothold in ferrous analysis is the polarograph.

*E. C. Pigott, "The Chemical and Physico-Chemical Analysis of Iron and Steel; Twelve Years' Advancement," *Metallurgia*, 1942, **26**, 75, 113, 145, 189, 202; **27**, 31, 80, 118.

In view of the rapid expansion of these techniques it is no matter for surprise that gravimetric processes, which at one time were considered to be superior to all others, are now gradually receding into obscurity.

Also conspicuous are the advances in control methods for determining oxygen and hydrogen, but some of the procedures are still elaborate and far from being within the scope of the average laboratory.

In choosing from the flood of methods with which the ferrous analyst is to-day confronted it is necessary to adhere to such guiding principles as reliability, simplicity, uniformity of technique, initial and operating costs, skill required, rapidity, amount of sample, and sources of error.

It is certain that mere novelty in a method is not enough. Rather is it desirable to select those methods that will fit most readily into the general laboratory scheme.

The inception of method standardisation by the Iron and Steel Institute was welcomed as an official effort to reduce dispute discrepancies; procedures for sulphur, phosphorus and lead have been devised by the Committee responsible, who emphasised that referee, rather than routine, procedure was the aim. The decision to evolve standard methods arose from a lack of uniformity in regard to detailed analytical procedure.

Authors are still at variance as to the best mode of presentation, especially in respect of the amount of detail. Unfortunately, there is little consistency in the interpretation of new methods and operational detail can, and does, vary widely from one laboratory to another. On the other hand, agreement of a high order is commonplace, suggesting that exhaustive descriptions may be bordering on dogmatic pedantry. The problem is a difficult one and calls for the utmost discrimination. The qualified operator can be relied upon to reliably assess the permissible latitude at the various stages in a method. Yet authors must consider even the reader intimidated by omission of the obvious. A useful guiding principle is to see that the permissible elasticity limits are clearly indicated throughout. An error of 10 mg. in weighing a lg.-sample for a carbon determination is of no consequence, yet a titration error of 0.1 ml. in the evaluation of an iron ore may vitiate the result by more than 0.1%. Conditions of evaporation are infinitely more critical in the separation of tungsten than in that of silicon. Such differences are well known, but those in new methods need to be emphasised. As ferrous analysis becomes increasingly advanced and specialised, clarity in presentation becomes more than ever important.

A careful count shows the rate at which papers have appeared to be something just over half the peace-time rate.

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Precision of measurement is at a maximum at the centre of the absorption range.
- Haywood. *Metallurgia*, 1942, 26, 117, 148.
Principles and applications of Spekker absorptometer.
- Johnson. *Iron Age*, 1946, 157, 66.
Nb, Ti, W by hydroquinone reactions.
- Pigott. *Iron and Steel*, 1944, 17, 285, 319, 363, 366.
Detailed consideration of photocells and instruments.
- Rogers. *Metallurgia*, 1945, 33, 13.
In 18 : 8 steels; mod. of Vaughan's Spekker methods for Cr, Mn, Mo, Ni.
- Silverthorn and Curtis. *Metals and Alloys*, 1942, 15, 245.
Cu and Mn; 15-min. accurate method entailing no preliminary sepn.
- States and Anderson. *J. Opt. Soc. Amer.*, 1942, 32, 659.
Effect of stray light on reading/concentration ratio.
- Stillman. *Proc. Amer. Soc. Test. Mat.*, 1944, 44, 740.
Alphabetical bibliography composed of refs. to papers in *Ind. Eng. Chem. Anal. ed.* 1–16 and *Analyst* 62–69.
- Vaughan. *Inst. Chem.*, Feb., 1942. Published lecture.
Ni by iodine—dimethylglyoxime method and Si as silico-molybdate with composite scheme for Cr, Mn, Mo, Ni, V.
- ### MICROCHEMICAL.
- Belcher and Spooner. *Metallurgia*, 1944, 29, 329; 30, 49.
Organic reagents.
- Hadfield. *Metal Treatment*, 1944, 21, 19.
Grav. and vol. procedures.
- Klinger et al. *Iron and Steel Inst.*, 1943. Translation No. 169.
Original in *Ange. Chem.*, 1940, 53, 537, and in *Tech. Mitt. Krupp. Forschung*, 1940, 3, 255.
Review. Al, C, Cr, Fe, Mn, Mo, Ni, N, P, Si, S, Ti, W, V. using variety of technique.
- Pigott. *Iron and Steel*, 1943, 17, 2, 140.
Abridged translations of papers by Benedicks and Treje, Leroy and Niessner followed by description of rapid quantitative photoelectric methods for C, Cr, Mn, Mo, P, S on a few mg. of sample. Numerous refs.
- Pigott. *Metal Treatment*, 1943, 10, 239, 272.
Photoelectric and polarographic applications and economics, with description and use of instruments.
- Sand. *Metallurgia*, 1944, 30, 107.
Micro-electrolysis.
- Vaughan. *Nature*, 1943, 152, 555.
Résumé of symposium: composite photoelectric method for Cr, Mn, Mo, Ni on a 4-mg. sample.
- ### POLAROGRAPHIC.
- Birdsall. *Steel*, 1945, 116, 122, 162.
Discussion and outlines of procedure.
- Buckley and Taylor. *Electrochem., Soc.*, 1945.
Discussion.
- Lykken et al. *Ind. Eng. Chem. Anal. ed.*, 1945, 17, 724.
Automatic photoelectric recording of polarograms.
- Sand. *Electrical Review*, 1942, 131, 297.
Theory, evolution and procedure.
- Sheet Metal Ind., 1942, 16, 619.
Detailed survey.
- Stackelberg et al. *Iron and Steel Inst.*, 1942. Translation No. 85. Original in *Arch. Eisenhüttenw.*, 1939, 13, 249.
- Thanheiser and Willems. *Iron and Steel Inst.*, 1942. Translation No. 83. Original in *Arch. Eisenhüttenw.*, 1939, 13, 73.
(Cr, Mo, V.)

SPECTROGRAPHIC.

- Ballard et al. *J. Opt. Soc. Amer.*, 1943, **33**, 667.
Bur. of Mines sampling procedure.
- Bardocz. *Spectrochim. Acta*, 1944, **2**, 350.
Cr, Mn, Ni, Si.
- Barker, Convey and Oldfield. *J. Iron and Steel Inst.*, 1941, 183P.
Cr, Mn, Mo, Ni, Si, V in segregates by single 15-sec. expos. using pointed silver electrode.
- Beard. *Sci. J.R. Col. Sci.*, 1944, **14**, 124.
Discussion; results of photographic methods dependent entirely on empirical data.
- Boettner and Brewington. *J. Opt. Soc. Amer.*, 1944, **34**, 6.
Using secondary emission photocells.
- Cameron. *J. W. Scot. Iron and Steel Inst.*, 1943, **51**, 15.
Review; steelworks. appl.
- Carlsson. *Iron and Steel Inst.*, 1946. Translation No. 267.
Original in *Jernkonterets Ann.*, 1945, **129**, 193.
- Carlsson. *Jernkonterets Ann.*, 1945, **129**, 193.
Interrupted D.C. arc method for Al, Cr, Co, Cu, Mo, Ni, Sn, Ti, V, when present within 0.1-0.001% range for which chemical methods are less accurate. Pure solutops, evap. on carbon electrode, preferred to chemically standardised steels. Accuracy $\pm 4-7\%$.
- Castro. *Rev. Met. Mem.*, 1942, **39**, 55, 84, 112.
Detailed survey.
- Coates et al. *J. Soc. Chem. Ind.*, 1942, **61**, 21, 62, 51; **63**, 83.
Plate calibration.
- Convey and Young. *J. Birm. Met. Soc.*, 1945, **25**, 168.
Discussion on excitation sources and stabilising of spectral emission.
- Cooper. *Analyst*, 1946, **71**, 356.
Survey of developments.
- Covey. *Metallurgia*, 1946, **34**, 331.
History and present status.
- Dietert and Schub. *Trans. Amer. Fdyment's Assoc.*, 1945, **52**, 889.
Description of equipment for rapid analysis in foundries.
- Ellis. *J. Opt. Soc. Amer.*, 1941, **31**, 534.
Lundegardh flame method.
- Girschig. *Rev. Met. Mem.*, 1943, **40**, 143, 175, 202, 252.
Apparatus, including automatic recording devices.
- Habitz. *Spectrochim. Acta*, 1942, **2**, 158.
Recommended lines for Cr, Mo, Ti.
- Hasler et al. *Ind. Eng. Chem. Anal. ed.*, 1943, **15**, 102.
D. C. arc and specially shaped carbon electrode for stainless steels.
- Hasler et al. *A.S.T.M. Bulletin*, 1946, Mar., 22.
Direct reading unit.
- Hammond and Fong. *J. Amer. Soc. Nav. Eng.*, 1943, **55**, 620.
Comparison with chemical procedure.
- Henderson et al. *J. Soc. Chem. Ind.*, 1945, **64**, 309.
Curved galvo. scale to eliminate calculations.
- Heyes. *Mitt K.W. Eisenfor.*, 1942, **24**, 1.
Mod. of Thanheiser and Heyes' method for SiO_2 and Al_2O_3 inclusions.
- Hurst and Riley. *J. Iron and Steel Inst.*, 1946.
Cast iron; all elements except C, P, S; Barker's flat surface technique; discussion; importance of layout.
- Irish. *J. Opt. Soc. Amer.*, 1945, **35**, 226.
Bethlehem Steel Co's methods as applied at weekly rate of 8,000 determ.
- Iron and Steel, 1942, **15**, 241.
(Segregates).
- Keck. *Spectrochim. Acta*, 1944, **2**, 389.
Automatic switching appar.
- Kincard. *J. Opt. Soc. Amer.*, 1944, **34**, 141.
Fabricated iron and steel.
- Ling et al. *Fdy. Trade J.*, 1943, **70**, 233, 240.
In cast iron.
- Matubona and Osida. *Nippon Kinyoku Gakkai*, 1941, **5**, 157.
Characteristic curve of photographic plate.
- McClelland. *Analyst*, 1946, **71**, 129.
Intermittent A.C. arc technique.
- Nitchie. *Steel*, 1942, **111**, 82, 124.
Steelworks appl.
- Norman and Johnson. *Ind. Eng. Chem. Anal. ed.*, 1945, **17**, 233.
Limits of detection for P (0.05-0.80 mg), Ti (0.04-1 mg.) and Zr (0.5-4 mg.)
- Nusbaum et al. *J. Opt. Soc. Amer.*, 1944, **34**, 33.
Briquetting procedure.
- Owen. *Iron Age*, 1946, **158**, 50, 61.
Time-saving modifications.
- Pochman. *Welding J.*, 1945, **24**, 564.
Development principles and appl. to castings, forgings, sheet bar, welding rods and welding beads.
- Post et al. *Ind. Eng. Chem. Anal. ed.*, 1945, **17**, 412.
Percentage atomic dilution in place of standards curves.
- Rozsa. *Iron Age*, 1943, **151**, 58.
Residual elements in O.H. steel; modern layout.
- Rozsa. *Iron Age*, 1946, **157**, 42.
Charging-floor spectrographic equipment; 7 elements in 15 minutes.
- Rohner. *Schweizer Arch.*, 1945, **11**, 311.
Rapid cine film technique.
- Rynninger. *Jernkonterets Ann.*, 1945, **129**, 1-31.
Cr, Cu, Mn, Mo, Ni, Si lines for Cr-Ni-Mo steel; spark excitation of low alloy steels has accuracy of 2.5-3%, this error being due principally to plate imperfections.
- Sample. *Amer. Inst. Min. Met. Eng. : Blast Furnace and Steel Plant*, 1942, **30**, 875.
O.H. bath sampling, determ. of Cr, Cu, Sn.
- Schliessmann. *Arch. Eisenhüttenw.*, 1941, **15**, 167.
Condensed spark.
- Selmi. *Heat-treating Forging (A.I.S.I.)*, 1941, **27**, 291, 304.
Accidental amounts of Al, Cr, Cu, Mo, Ni, Si in cold-drawing steels.
- Sherman and Jenkins. *J. Amer. Soc. Nav. Eng.*, 1943, **55**, 189-312.
Comprehensive review.
- Shirley and Elliot. *J. Iron and Steel Inst.*, 1943.
Steelworks appl. Modn. designed to minimise maximum errors.
- Steel and Johnston. *J. Soc. Chem. Ind.*, 1945, **64**, 278.
Flat surface technique with copper counter electrode for Al, Cr, Mn, Ni, Si, in 18-8 steels; accuracy $\pm 0.47\%$ Cr; $\pm 0.30\%$ Ni.
- Smith. *Metal Ind.*, 1945, **67**, 226.
Description of Russian methods.
- Smith. *Fdy. Trade J.*, 1945, **75**, 55.
Discussion.
- Smith. *Fdy. Trade J.*, 1946, **78**, 311.
Fundamentals.
- Taylor. *J. Sci. Instruments*, 1942, **19**, 11.
Device to minimise wandering of the arc.
- Tippett. *Iron and Steel*, 1943, **16**, 224.
Moderate equipment for steelwork.
- Vincent and Sawyer. *J. Opt. Soc. Amer.*, 1942, **32**, 686.
Use of statistical methods to enhance precision.
- Wehrich and Schwarz. *Arch. Eisenh.*, 1941, **15**, 33.
Segregates.
- Wolfe and Fowler. *J. Opt. Soc. Amer.*, 1945, **35**, 86.
Trace elements.
- Young. *Engineer*, 1944, **178**, 116.
Single spectrogram from six superimposed exposures to overcome heterogeneity errors.

MISCELLANEOUS.

- Arnold. *Steel*, 1943, **110**, 74.
Electrographic principles applied to alloy components in steel; survey of literature.
- Evans and Higgs. *Analyst*, 1945, **70**, 75.
System of spot tests for Al, Cr, Co, Pb, Mn, Mo, Ni, Ti, Se, W, V in alloy steels.
- Fuche and Möhrle. *Arch. Eisenhüttenw.*, 1944, **18**, 47.
Drop reactions for Al, Cr, Cu, Mo, Ni, Ti, W, V.
- G. Fred. Smith Chem. Co., Ohio. *Cerate Oxidimetry*, pp. 123, 1942.
- G. Fred. Smith Chem. Co., Ohio. *Colorimetry for Chemists* pp. 133, 1945.
- G. Fred. Smith Chem. Co., Ohio. *Phenanthroline*, pp. 103, 1944.
- Jessop. *Iron Coal Trades Rev.*, 1944, **146**, 641.
Potentiometric and polarographic analysis of solutions.
- Kriesel. *Metall und Erz.*, 1942, **39**, 143.
Vol. determ. of Cr, Fe, V in slags, ores and ferro-vanadium.
- Pigott. *Iron and Steel*, 1942, **15**, 196, 202.
Rapid qualitative tests for iron, steel and foundry materials. (Al, As, Be, B, C, Cr, Cu, Fe, Pb, Mn, Mo, Ni, Nb, P, Se, Si, S, Ta, Te, Sn, Ti, W, U, V, Zn, Zr).
- Pigott. *Metallurgia*, Sept., 1941.
Survey of qualitative grav. and col. reagents for Al, As, B, Be, Cr, Co, Cu, Fe, Mo, N, Nb, Se, P, Ta, Te, Ti, V, W, Zn, Zr. 118 Refs.
- Powell. *Iron and Steel*, 1943, **16**, 428, 468, 477.
Recent apparatus.
- Steel, 1943, **112**, 128, 148.
Amer. I.S. Inst. standard sampling methods.
- Steinmetz. *Ind. Eng. Chem. Anal. ed.*, 1942, **14**, 109.
Small amounts of Al, Mg, Ti and V by large-cell mercury cathode method.
- Thanheiser and Waterkamp. *Iron and Steel Inst.*, 1942. Translation No. 95. Original in *Arch. Eisenhüttenw.*, 1941, **15**, 129, and *Mitt. K.W. Eisenfor.*, 1941, **23**, 81.
Al, Cr, Cu, Mn, Mo, Ni, P, Si, S, Ti, V and W semi-quantitatively in steel by non-defacing drop reactions.

Van Valkenburg and Crawford. *J. Ind. Eng. Chem. Anal. ed.*
Detection of Cr, Co, Ni, Mn, Mo, Fe, Ti, U, V, W in ores, after fusion with Sn ,
hypophosphite and extraction with H_2O or H_2O_2 .

Weirich and Schwertner. *Arch. Eisenhüttenw.*, 1942, **16**, 45.
Spot tests for Co, Fe, Ni.

Part 2. Papers dealing with the determination of single constituents.

Aluminium

The mercury cathode process has been developed to serve for minute amounts. In adapting the compiler's oxine method to a rich nickel-chromium steel, a preliminary removal of the chromium was resorted to by Steele and Russell. A direct procedure advocated for this alloy by the compiler is more rapid. Details have been given of a new aluminum method. One of three spectrographic processes is preceded by a mercury cathode separation.

MERCURY CATHODE SEPARATION.

Hammarberg and Phragmén. *Jernkonterets Ann.*, 1943, **127**, 608.

Review: Method for minute amounts; sepn. of Mn as phosphate; of Fe, Ti, V finally by cupferron; of Si by HClO_4 . Al weighed as AlPO_4 .

WITHOUT PRE-SEPARATION OF IRON.

Pigott. *Iron and Steel*, 1943, **16**, 325.

In 25% Ni—10% Cr—1% Al steel by direct 8-hydroxyquinoline pptn. from cyanide-tartrate solution.

Steele and Russell. *Iron and Steel*, 1943, **16**, 182, 200.

In 25% Ni—10% Cr—1% Al steel by modification of Pigott's method (*J. S. Chem. Ind.* 1939, **68**, 139). Modification consists in first eliminating Cr by volatilization as chromyl chloride and in re-pptn. under special conditions to overcome Ni interference.

WITH PRE-SEPARATION OF IRON.

Box. *Analyst*, 1946, **71**, 317.

In steel. Hydroxides of Al, Cr, Ti, Zr (and some Fe) pptd. AmOH ; Fe into complex cyanide; Al, Ti, Zr pptd. oxine; Ti, Zr removed cupferron.

COLORIMETRIC.

Craft and Makepiece. *Ind. Chem. Anal. ed.*, 1945, **17**, 206.

Determ. of 0.4/1.5% by means of aluminum.

FLUOROMETRIC

Weissler and White. *Ind. Eng. Chem. Anal. ed.*, 1946, **18**, 530.

0.001–1.0%; aliquot parts of a macro sample; interfering ions removed by electrolysis in a Hg-cathode cell.

SPECTROGRAPHIC.

Carlsson. *Iron and Steel Inst.*, 1946, Trans. No. 290 Original in *Ann. Jernkonterets*, 1942, **126**, 161.

Carlsson. *Jernkonterets Ann.*, 1942, **126**, 161.

Arc excitation method for low Al_2O_3 contents in steel.

Casteo. *Metal Treatment*, 1946, **13**, 182.

Contents 0.004–0.025% by spark technique.

Hammersburg and Phragmén. *Jernkonterets Ann.*, 1943, **127**, 608.

Spiers et al. *Ind. Eng. Chem. Anal. ed.*, 1945, **17**, 772.

Electrode absorption of one drop of soln. obtained by mercury cathode sepn. applied to 20–500 mg. of sample.

Arsenic

No new methods were published for determining arsenic in iron and steel, but estimation of the element in ferro-tungsten has received consideration.

Weirich and Haas. *Arch. Eisenhüttenw.*, 1942, **16**, 129.

In ferro-tungsten: treatment with HNO_3 and HF preceded by Na_2O_2 —fusion to obviate partial volatilization.

Boron

Two new colorimetric procedures have been devised for routine application, and sensitive spectrographic methods described.

COLORIMETRIC.

Rudolph and Flickinger. *Steel*, 1943, **112**, 114, 131, 149.

Routine method exploiting blue quinizarin reaction in H_2SO_4 .

Weinberg et al. *Ind. Eng. Chem. Anal. ed.*, 1945, **17**, 419.

Rapid routine method for 0.001/0.005% B. in corrosion, resistant steels.

SPECTROGRAPHIC.

Corlias and Scribner. *J. Res. Nat. Bur. Stand.*, 1946, **38**, 351.

$\frac{1}{2}$ in. dia. electrodes; high voltage A.C. arc; amounts down to 0.0006%.

Nat. Bur. Stand. Tech. News Bull., 1946, **348**, 27.

In steel; three methods, one of which serves for amounts down to 0.0001%.

Falkova. *Comptes Rendus de l'Acad. des Sci. de l'URSS*, 1945, **48**, 179.

In filings, 0.2 g. of which placed in Al; A.C. arc; iron rod used as other electrode.

Carbon

Most investigations have been concerned with combustion procedure, including low-pressure combustion. Gravimetric handling of the evolved carbon dioxide has been compared with volumetric, while one very accurate process entails electrical conductivity measurements of the absorbent and another, condensation in liquid air. Attention has been paid also to the direct determination of combined carbon. A novel method assesses the carbon content on a basis of hardness values. Rapid bath-sample analysis has undergone development.

HIGH TEMPERATURE COMBUSTION IN OXYGEN.

Cotton. *Analyst*, 1945, **70**, 466.

Grav. and vol. finish compared; use of evacuated bottles charged with $\text{BaCl}_2/\text{NaOH}$ absorbent.

Ericsson. *Iron and Steel Inst.*, 1945, Translation No. 232.

Original in *Jernkonterets Ann.*, 1944, **123**, 579.

Ericsson. *Jernkonterets Ann.*, 1944, **123**, 579.

CO_2 absorbed in $\text{Ba}(\text{OH})_2$ soln. and change in electrical conductivity measured by means of bridge consisting of rheostat, oscillator and cathode-ray tube. Accuracy $\pm 0.0005\%$.

Gurry and Trigg. *Ind. Eng. Chem. Anal. ed.*, 1944, **16**, 248.

Low-pressure combustion.

Locke. *Iron Age*, 1945, **117**, 69, 176B.

Electric furnace bath-sample methods reviewed.

Murray and Ashley. *Ind. Eng. Chem. Anal. ed.*, 1944, **16**, 242.

Low-pressure combustion.

Saxer et al. *Blast Furnace and Steel Plant*, 1941, **29**, 718.

Precision factors discussed.

Sheet Metal Ind., 1942, **16**, 1488.

Improved Ströhlein type apparatus.

Steel, 1944, **115**, 93.

With S; determinations for C (and S) in steel, iron, coal, coke, etc.

Wootton and Guldner. *Ind. Eng. Chem. Anal. ed.*, 1942, **14**, 835.

In low carbon iron and steel by low-pressure combustion.

Zhuravleva and Chufarov. *Zavod Lab.*, 1940, No. 5, 6, 498.

With S; at $1,300^\circ\text{C}$; $\text{CO}_2 + \text{SO}_2$ condensed in liquid air, evolved, and CO_2 re-condensed.

SPECTROGRAPHIC.

Blank and Sventitsky. *Comptes Rendus de l'Acad. des Sci. de l'URSS*, 1945, **48**, 252.

Use of lines CuI 4267 and CuII 3296–89.

MICROCHEMICAL.

Bayliss et al. *Ind. Eng. Chem. Anal. ed.*, 1943, **15**, 68.

Occasional micro-combustion method for low carbon steel; modification of standard carbon-hydrogen train as used for organic substances.

Ingram. *Metallurgia*, 1945, **32**, 137.

With S by micro-combustion.

MISCELLANEOUS.

Donaldson. *Trans. Amer. Fdyman's Assoc.*, 1943, **50**, 931.

Combined carbon in iron and steel; soln. in $\text{HNO}_3 + \text{H}_2\text{SO}_4 + \text{H}_3\text{PO}_4 + \text{AgNO}_3$ treated with $\text{Am}_2\text{S}_2\text{O}_8$ and evolved gases passed over heated CuO , with absorption of CO_2 in soda asbestos.

Kern. *Amer. Fdyman's*, 1942, **4**, 8.

Electric furnace bath-sampling procedure; ref. curve relating Brinell hardness to carbon content; spoon samples killed with Al and water-quenched. Suitable for low alloy steels containing 0.15/0.45% C.

Stanley and Yensen. *Ind. Eng. Chem. Anal. ed.*, 1945, **17**, 699.

20-min. method for amounts up to 0.01% in soft magnetic alloys.

Wentzel and Riott. *J. Amer. Soc. Metals*, Oct., 1941.

Discussion of magnetic induction methods.

White. *Foundry*, 1946, **74**, 109.

In pig iron; Si and graphitic carbon contents have linear relationship.

Chromium

Titrimetric methods have received little attention. In a colorimetric procedure, perchloric acid is utilised not only as a means of suppressing the colouration of ferric salts but also to intensify the dichromate colour. The American photoelectric "Photometer" designed by Sheard and Sanford and used in one of the methods is one of the earlier one-cell instruments and employs a photovoltaic photocell and constant voltage transformer. A noteworthy development is the spectrographic determination of stainless steel contents.

VOLUMETRIC.

Tomb. *Iron Age*, 1946, **157**, 64.
In high chromium steels.

COLORIMETRIC.

Singer and Chambers. *Ind. Eng. Chem. Anal. ed.*, 1944, **16**, 507.
Ordinarily specific method for contents up to 1% in steel; based on intensification of colour of dichromate by ferric perchlorate.

PHOTOMETRIC.

Jacoby. *Foundry*, 1944, **72**, 111, 178.
Appl. of Sheard-Sanford "Photometer."

SPECTROGRAPHIC

Couliette. *Ind. Eng. Chem. Anal. ed.*, 1943, **15**, 732.
In stainless steels.

MISCELLANEOUS.

De Lipa. *Analyst*, 1946, **71**, 34.
In low alloy steels; 20-min. method.

Cobalt

The emphasis has been on photometric procedure, classical methods such as the zinc oxide-nitroso- β -naphthol process having been accorded neither modification nor development. The new methods are satisfactorily immune from nickel interference.

PHOTOMETRIC.

Bogatzki. *J. Iron and Steel Inst.*, 1945. Translation No. 221.
Original in *Arch. Eisenhüttenw.*, 1943, **17**, 125.
Direct method for tool and high-speed steels.

Clarke. *Iron Age*, 1942, **150**, 45.
Soln. in HCl treated with SnCl_2 ; no interference from Ni; Fisher photoelectric photometer.

Haywood and Wood. *J. Soc. Chem. Ind.*, 1943, **62**, 37.
Reaction with nitroso-R-salt, with excess of HNO_3 to inhibit interference from Ni.

MISCELLANEOUS.

Evans. *Analyst*, 1943, **68**, 67.
Cobalt: cyanide sepn. from ferric iron.

Copper

New literature devoted to the determination of this increasingly important constituent reflects the growing preference for colorimetric technique, but three electrolytic methods have been evolved.

COLORIMETRIC.

Clardy et al. *Ind. Eng. Chem. Anal. ed.*, 1945, **17**, 791.
15-min. method.

Thomas. *Proc. Amer. Soc. Test. Mat.*, 1944, **44**, 769.
In both iron and steel by means of diethylthiocarbamate reaction.

PHOTOMETRIC.

Haywood and Wood. *Analyst*, 1943, **68**, 206.
Diethylthiocarbamate method for iron and steel with separation of nickel; Spekker absorptiometer.

Milner. *Ind. Eng. Chem. Anal. ed.*, 1946, **18**, 94.
In corrosion-resistant steels: 12 determ. in two hours after soln.

Quandel. *Arch. Eisenhüttenw.*, 1941, **14**, 601.

ELECTROLYTIC.

Levine and Seaman. *Ind. Eng. Chem. Anal. ed.*, 1944, **16**, 80.
Pt.-gauze cathode.

Silverman et al. *Ind. Eng. Chem. Anal. ed.*, 1942, **14**, 236.
Deposition from H_2SO_4 - H_3PO_4 followed by cyanometric determ. of Ni.

MICROCHEMICAL.

Macnevin and Bournique. *Ind. Eng. Chem. Anal. ed.*, 1943, **13**, 757.
In cast iron by microelectrolysis.

To be continued.

Comparison of Electro-Plated Finishes under Humidity Tests

Communication from Mr. E. E. Halls

APPRECIATION must be expressed for the durability test results that Mr. Frank Taylor enabled to be published in the December issue of *METALLURGIA*, pp. 63-64. Concisely presented, they are self-explanatory, and carry a wealth of information in a small space. The results are particularly useful in that the processes represent commercial working, and not merely laboratory scale operation, and again insofar as the components are an awkward box-shaped type. The photographs reproduced in all fairness depict the performance of the inside and outside of the boxes for each finish tested; practical men and technicians alike appreciate the significance of this feature.

The liberty is taken of commenting upon the data presented and it is hoped that the remarks made will be regarded as complimentary and constructive, and in no sense of a critical nature.

In conjunction with thickness, the weight of the coating has to be considered (i.e., density of the metal

deposited). This is necessary from the points of view of economics (plating time and cost of metal deposited) and durability (i.e., quantity of metal available for sacrificial corrosion). The attached curves include the metals discussed and show the relationship to close approximation.

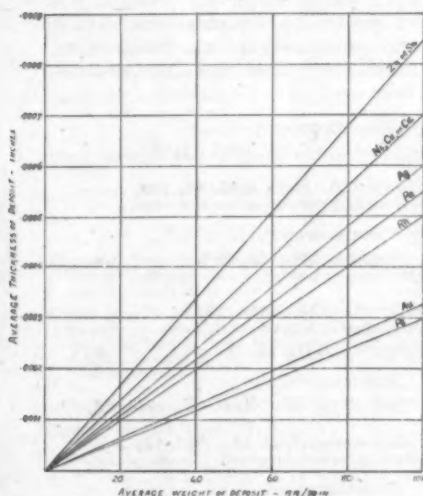
The excellent performance of passivated cadmium (Fig. 1) is particularly stressed in relation to the average thickness of 0.0005 in., which is the thinnest plating shown.

Secondly, the performance of passivated (chrome) zinc in Fig. 3, is considered of high merit, although the coating is thicker, average 0.0008 in.; the weight of this coating is, however, of similar order to that of the cadmium shown in Fig. 1.

The composite nickel/tin and nickel coatings are comparatively very heavy (Figs. 6 and 7), and the tin in Fig. 8 is very heavy; none of these performed

nearly so well; the lighter tin coatings naturally fared worse (Figs. 9 and 10).

Often coating of restricted thickness are essential for dimensional or electrical reasons. Electronegative coatings of nickel, tin, copper and the like are useless for steel because they are too porous or non-continuous to yield a moisture-proof covering. Electropositive coatings of zinc or cadmium become essential. Mr. Taylor's work again stresses that with such coatings, of medium thickness, the chromate passivation adds to the merits of the coating that immunity from corrosion that nickel or copper possesses. Hence, the chromate



Relation between average weight and average thickness of electro-coatings of zinc, nickel, tin, copper, cadmium, silver, gold, palladium, rhodium and platinum.

film gives value out of all proportion to the cost and effort for its provision.

In the finishing of small precision-made components of complex shape, for instruments, light electrical and radio apparatus, dimensions are all important. This applies not only to screws and nuts, tapped and clearance holes, but also to numerous other pieces, or portions

of pieces, i.e., cams, levers, pawls, pillars, clamp plates, armatures, magnetic cores and yokes, etc. These industries are fully aware of the importance of the thickness of coating. They generally dimension their components to allow for the average thickness of plating and to take care, at least to some degree, of build-up at prominences, edges of parts, etc. Even so, in dealing with small work, a compromise has to be achieved in many cases. Variation in thickness of plating occurs not only over the same component, but also from one part to another in a vat load. This is especially marked with industrial plating where the plant is always fully loaded and has to cater for thousands of different shapes during the week. A variation from component to component of 1 to 2 for the range from min. to max. has to be allowed under the best of such conditions. These limits have, therefore, to be borne in mind when settling a minimum and an average value on a single component, especially as the maximum figure is the one that concerns the assembly shop, and perhaps the magnetic or electrical tester after assembly.

For the general type of work in these industries, where zinc is used as the protective coating on steel, the average limiting values for the zinc on any single component are usually minimum 0.0005 in., maximum 0.0010 in. The minimum local thickness (on any point on a component that can be contacted with a 1 in. diameter sphere, and is more than $\frac{1}{4}$ in. from any edge) is 0.0003 in. For the special work where dimensions or other requirements enforce it, the average thickness limits often used are 0.0003 in. minimum and 0.0006 in. maximum. A minimum local thickness of 0.0002 in. can be applied but is not generally found necessary in practice. The reason for this is that the components are nearly always very irregular in shape, with bends, holes and the like, and rarely is there a point contactable with a 1 in. diameter sphere in the above sense. Further, using a cyanide zinc solution, uniformity to ensure the suggested minimum local thickness is achieved.

COMPARATIVE BEHAVIOUR OF RELATIVELY THIN ELECTRO-PLATE COATINGS, WITH AND WITHOUT PASSIVATION, ON SOFT IRON, WHEN SUBJECTED TO CYCLES OF K.III TEST.

Finish	(1) Zinc and thin Nitrocellulose Lacquer	(2) Zinc	(3) Zinc, chromate passivated	(4) Cadmium	(5) Cadmium, chromate passivated	(6) Nickel	(7) Nickel with chromium flash
Thickness of electro-plate finish:—							
Minimum average	0.0003 in.	0.0003 in.	0.0003 in.	0.0003 in.	0.0003 in.	0.0004 in.	0.0004 in.
Maximum average	0.0006 in.	0.0006 in.	0.0006 in.	0.0006 in.	0.0006 in.	0.0008 in.	0.0008 in.
Performance under Wet Test:—							
1st cycle	Slight white corrosion products general.	White corrosion products general.	Unaffected.	Darkening in colour.	Unaffected.	A few rust spots developed.	Trace of rust developed.
2nd "	White corrosion, more prolific.	White products, more prolific.	A tendency to form slight white corrosion products in a few local patches.	Colour darker and a few rust spots apparent.	Unaffected.	Rusting developed a little further.	Still only one trace of rusting.
3rd "	Progressive whitening.	Progressive whitening and a trace of rust in a bend.	Tendency to white corrosion at edges, but very slight.	Rust spots more pronounced.	Unaffected.	Rust spots fairly general over all surfaces.	Little further deterioration.
4th "	Progressive whitening, no rusting.	White corrosion much heavier.	Whitening still very slight and confined to edges.	Rust along most edges and rust spots on surfaces more pronounced.	Unaffected.	Rust spots general and heavy.	Traces of rust on edges.
21st "	Heavily corroded to white products but only a few rust spots.	Heavily corroded to white products, traces only of rust.	50% of surfaces deteriorated to a slight white film. No rusting. Condition exceedingly good.	Little marked change. Condition dark and dirty in appearance. Rust on edges and a number of rust spots on most surfaces.	A little darkening in colour, otherwise unaffected. Condition excellent.	Rusting very heavy and condition exceedingly poor.	Rusting general in the form of spots and patches. Some patches very severe. Conditions much superior to that of the nickel finish (6).

Using cadmium, somewhat similar arguments hold good for the adoption of these limits.

The object in presenting these very brief notes on thickness of coating, is to give emphasis to the desirability of applying the chromate passivation. This treatment does not enable less zinc or cadmium to be used, but it does protect that which it is possible to apply and thus indirectly ensures a prolonged serviceability.

Regarding nickel, it is agreed that anything less than 0.001 in. minimum thickness is almost useless on iron and steel. Tin is in a similar category. Nickel of course, is improved by a chromium flash which apparently gives passivation in some manner not clearly understood, so that rusting is inhibited. It is interesting to note that for critically designed relays where zinc and cadmium are impossible for use on pole pieces, armatures and yokes on account of "mushing," i.e., powdering under impact or wear on the rapidly moving areas, and pounding to a "mush" that causes sticking in operation, as well as on account of causing loss of efficiency in the magnetic circuit, nickel has to be chosen for the protection of these soft iron parts. To ensure relay operation, a limit still has to be imposed. on the maximum nickel thickness in many cases, and average thickness limits for single components typical of those adopted are 0.0004 in. minimum, 0.0008 in. maximum. Apparatus of this type often has to function in the equivalent of damp tropical climates, and manufacturers do achieve appreciable enhancement of the protective value of the coating by a final flash coat of chromium.

Finally, typical test results taken on iron parts of this category, with these finishes, are informative, and are given in the attached table. These results were taken on components from production, plated to the limits specified, not specially plated to precise values. The test cycle was to W.T. Board Spec. K 110, including a preliminary dry cycle (6 hours at 71° C.), followed by successive wet cycles (6 hours at 60° C., 100% humidity, remaining in the humid atmosphere with condensation for the remainder of the 24 hours). The test results are self explanatory. The chromate passivated samples of zinc and cadmium plating are outstandingly good, especially the cadmium. This is particularly gratifying because these thin coatings are rather below the thicknesses that one would wish to adopt for such severe conditions. The zinc coatings all were the normal mat type from a cyanide electrolyte. "Bright" zinc with passivation would have performed even better. Concerning the nickel coatings, it can be seen that the chromium flash gives marked improvement, but not sufficient for justifying the use of such thin nickel coatings in severe tropical atmospheres. At the same time, it is admitted that considerations other than those of corrosion resistance enter, and they cannot be overlooked. Often a compromise must be made to suit the specific problem.

Comments by Mr. Frank Taylor

IN presenting a comparison of electro-plated finishes under humidity (K110 test) in our last issue, it was taken for granted that readers would assume that the results given were merely a précis of the work carried out. The thickness of deposits applied was based on figures

found suitable for good corrosion protection and applied by normal commercial methods. When assessing these thicknesses it was necessary to consider such factors as weight of coating, porosity, corrosion and erosion resistance, whether deposit was electropositive or electronegative in relation to base metal, stress and ductility of certain deposits, etc. The thickness figures used were the result of a large number of corrosion tests carried out by independent investigators throughout the country, who collectively, either via a Government Department or the British Standards Institution, have stipulated such thicknesses.

The classification of coatings with regard to their being electropositive or electronegative is rather misleading, because there are two schools of thought. In the latter classification the more electronegative metal becomes the anode in the electrolytic couple set up. According to this classification, the reverse classification to that employed by Mr. Halls occurs, viz., copper, nickel and tin are electropositive and zinc and cadmium electronegative. This is only a minor point but it leads to considerable confusion on the part of the layman, as he normally assumes that a more electronegative metal is the cathode in the electrolytic couple set up, whereas this may or may not be true according to the classification.

It is my opinion that a thin flash of chromium improves the corrosion resistance of the underlying nickel for two reasons:—

1. A comparatively large decrease in porosity of the composite nickel-chrome coating as compared with nickel alone. This can be quickly proved by the ferroxyl test.

2. A semi-passivation effect on the underlying basis metal, via any pores in the nickel deposit.

The comments by Mr. Halls are a corroboration of many of the results I have obtained. His chart of the relative behaviour of thin electrodeposited coatings under the K.110 test is comprehensive and further amplifies the advantages of the chromate passivation process. This fact is, to a large extent, being overlooked, and is to be deplored now that cadmium is being increasingly specified. Whether this is due to ignorance or to misinformation, it is not possible to say, but, like Mr. Halls, I am an earnest believer in the value of this process where maximum corrosion resistance is required on both cadmium and zinc coatings. The process is simple to apply and the cost negligible, and it ensures the best type of deposit with the minimum thickness.

Errata

On page 102 of December, 1946, issue, paragraph 4, of column 1, where it states: "where zinc was the only home produced metal"; this should read: "where zinc and aluminium were the only home produced metals."

On page 103 of the same issue, paragraph 3 of the section under the sub-heading "Zinc in Paints." The words: "the latter by co-fuming of lead sulphate and lead oxide" should read: "The latter by co-fuming of lead sulphate and zinc oxide."

On page 105 of the same issue, under the sub-heading "Procedure," line 19, "Cool filtrate and reserve for fractionation." should read: "Cool filtrate, dilute to 120 ml. and reserve for fractionation."

Some Recent Appointments and Staff Changes

Mr. LOUIS RIPLEY, O.B.E., has retired from his position as Principal Surveyor for Steel to Lloyd's Register of Shipping after 27 years' service. Mr. Ripley, who is well-known in the steel industry in this country, and in Middlesbrough in particular, received his early training in Sheffield. He was appointed to the staff of Lloyd's Register in 1919 and was stationed in Glasgow. On his being appointed Principal Surveyor for Steel, the London Committee decided that for geographical reasons he should be stationed at Middlesbrough. He has travelled extensively to all steel-producing centres in this country and is equally wellknown on the Continent.

To mark the occasion and as a token of their high regard for him, his colleagues at Middlesbrough and twenty-two other associates in the United Kingdom and the Continent co-operated in presenting him with a cheque.

Mr. W. E. LEWIS, B.Sc., has succeeded Mr. Ripley as Principal Surveyor for Steel. Like Mr. Ripley, he has travelled extensively to steel producing centres and is well-known in steel industries in this country and on the Continent. He received his early training at Ebbw Vale.

Mr. ELLIS HUNTER, in response to the unanimous request of the Council of the British Iron and Steel Federation, has agreed to stay in office as President for another year. Mr. Ellis Hunter is Deputy-Chairman and Managing Director of Dorman, Long and Co., Ltd.

Mr. C. S. GILL was re-elected Chairman and Mr. F. A. MARTIN elected Vice-Chairman of the British Steel Founders' Association for this year, at the Annual General Meeting of the British Steel Founders' Association recently held in Sheffield.

Mr. E. T. JUDGE, M.A., Chief Engineer, Dorman, Long and Co., Ltd., has been appointed a Director of the Company. Mr. Judge joined the Company as a trainee in 1930 on leaving Cambridge, where he obtained a Mechanical Science Tripos, with Honours. Following experience in the Central Engineering Department, he became Chief Technical Engineer to the Company in 1937. In 1944 he was appointed a Special Director. Mr. Judge is familiar with Iron and Steel Plants in this country, in pre-war Europe, and the United States of America where before and during the war he studied the latest developments.

It will be his task to carry through the design and construction of the Dorman, Long £8 million project for a new steel works and universal beam mill on the Lackenby site.

Mr. D. C. GUNN has been appointed Director of Research of the Incandescent Group of Companies. Until recently he was Manager, Engineer and Secretary of the Yorkshire Industrial Gas Centre Ltd.

Mr. Gunn has had considerable experience in the application of thermo engineering throughout the metallurgical and other industries, and research and development connected with the industrial utilisation of town gas. He is a graduate of London University and holds the M.Sc. degree in Gas Engineering of the Leeds University.

Mr. E. K. SCOTT has been appointed a Special Director of Dorman, Long and Co., Ltd. Mr. Scott, who is 42, returned recently from a 10 weeks' visit to China as a member of the British Government Goodwill Trade Mission. He knows the Far East well. In 1929 he went to Manchuria and represented the Company in China from 1932 to 1938. Shortly before the war he undertook a mission to Turkey for the Company. He saw service in France and went to Singapore as a Lieut.-Col., R.E.M.E., being transferred after the evacuation to General Wavell's staff in Java. On his way to Java he was captured by the Japs and held prisoner until the end of the war.



Mr. H. H. Richardson of Montreal has been appointed president of Aluminium Laboratories Ltd. and elected to the board of directors of the Aluminium Company of Canada Ltd. As president of Aluminium Laboratories Limited, he will direct the research and development activities undertaken for the Aluminium Limited group of companies in research laboratories at Arvida and Kingston in Canada and Banbury, Eng-

land. A graduate of Yale University in metallurgy, Mr. Richardson has spent 22 years in the aluminium industry in Canada, Great Britain and Switzerland.

Mr. KENNETH HALL has been appointed Managing Director of Northern Aluminium Co., Ltd. He recently resigned the Managing Directorship of the Indian Aluminium Company to take up this new appointment. Mr. S. E. CLOTWORTHY has also been appointed to the Board of this Company, which now consists of Mr. Kenneth Hall, Mr. D. Cannon Brooks, Mr. H. C. Thomas and Mr. S. E. Clotworthy.

Mr. STEPHEN L. FINCH has been appointed foundry manager of the Yorkshire Steel Foundry of Catton & Co., Ltd., Hunslet, Leeds. Mr. Finch was formerly connected with Leyland Motors, Limited, and more recently with K. and L. Steelfounders and Engineers, Ltd., Letchworth.

Mr. LAURENCE S. ROCKEFELLER, of New York, has been elected Director of the Board of the International Nickel Company of Canada Ltd., to fill the vacancy caused by the death of Mr. Thomas Morrison of Pittsburgh. His election was announced by Mr. Robert C. Stanley, Chairman and President of the Company, at the recent Board Meeting held this month.

Mr. Rockefeller is also a Director of the Rockefeller Centre Inc., Eastern Air Lines Inc., McDonnell Aircraft Corporation, The Merchants Fire Assurance Corporation, New York, and is a member of the New York City Airport Authority.

The Centrifugal Casting of Brass (63 : 37)

Ingots for Subsequent Cold Rolling

By T. B. Crow,* Ph.D., F.R.I.C., F.I.M.

This paper is a reproduction of a report, hitherto unpublished, of an experimental investigation (carried out with a full-sized specially constructed "production" unit) into the possibility of casting brass (63 : 37) "ingots" for subsequent cold rolling into finished strip.

Introduction

SOME twelve years ago the author began a course of experimental work, with the aid of a specially designed centrifugal casting machine. After many trials under all sorts of conditions of operational technique, a considerable degree of success was obtained; such success extending as far as satisfactory reports from users of the rolled strip originating from the process. In 1937, a Report was written and probably would have been published had not the outbreak of war rendered it necessary to discontinue the work.

During the last decade there has been a considerable addition to the literature on centrifugal casting, and papers, notably by Northcott, Hurst and Howson have dealt with various aspects of the process or with the properties of the resulting castings. It is noteworthy, however, that there has been little or no report of investigations which had, as their aim, the production of spun rings for cold rolling, and it is possible that the work of the present author is original in this respect. For this reason there appears to be some justification for its publication. Accordingly, the 1937 report is subjoined in practically its original form and the author gratefully acknowledges the permission of the directors of Emery Brothers Ltd., and of Messrs. Sheepbridge Stokes Centrifugal Castings Co., Ltd., for their kind permission to release it.

REPORT as at MARCH, 1937

(The Centrifugal Casting of Brass Ingots for subsequent Cold Rolling.)

Contents

- I. Objects of the Research.
- II. Description of the Plant.
- III. The Casting Process.
- IV. Examination of the Castings.
- V. Machining the Castings.
- VI. Rolling the Castings.
- VII. Tests on Finished Strip. The Future.

I. Objects of the Research

The experience of several firms engaged in the casting and cold rolling of brass (63 : 37) strip appears to be that there is considerable difficulty in producing rolled strip free from surface defects, in widths much greater than eight inches. The commonest surface defect is "spill" and its occurrence may necessitate the scrapping of a high proportion of output tonnage either by the manufacturer of the strip, or, by the user, for such defect frequently escapes detection until the sheet

metal is fabricated into finished articles. Since this defect arises during the casting process (in cases where the usual practice of casting into ordinary cast iron strip moulds is carried out) it follows that some modification of this process is desirable in order to eliminate it. Thus arises the fundamental object of this Research. A centrifugally cast ingot would have the shape of a table napkin ring and it would be cheaper to skim off the outside and inside surfaces of such a shaped ingot in a lathe or boring mill than to plane off the surface defects of a rectangular-shaped slab. There would also be other advantages, viz., (1) the heterogeneity and coarseness of the grain structure of a strip ingot would be replaced by a more uniform and smaller grain structure which should result in a tougher metal which would withstand a greater initial "reduction" by rolling, without cracking along the edges, before the first anneal; (2) a slab of brass 36 in. \times 14 in. \times 1 in. would weigh about 150 lbs., whereas the same weight of metal cast into a ring of, say, two feet diameter and fourteen inches in height would produce a casting of about $\frac{3}{4}$ in. thick; that is, one rolling and annealing operation would be eliminated; (3) it would appear more economical to roll a strip fifteen inches wide, and when finished or nearly finished (as regards the gauge required) to shear it to three widths of five inches than to produce the same weight of the same width and gauge of strip as three chill cast slabs of some five inches in width; (4) if 500 lb. of metal were to be cast into a ring fifteen inches wide, it would be about $1\frac{1}{2}$ in. thick, and if such a ring were rolled in one width, or in several widths, in a direction parallel to its circumference (see Figs. 1 and 3), the length of the finished strip in any gauge would be some three times that of an ordinary one-inch thick ingot; a notable advantage where the strip is to be used in automatic self-feeding presses; (5) it was expected that economies in personnel and in the melting shop would result; (6) a finished product, guaranteed free from hidden surface or internal defect, and having cold pressing or stamping properties at least equal to those of brass cast by ordinary methods, could be offered to the customer, who would thus be saved the loss of rejections after bearing the cost of fabrication.

It was, therefore, decided to carry out exhaustive experimental trials of the process on a sufficiently big scale to enable reliable conclusions to be formed both as regards its metallurgical and its economic soundness. This meant that the unit must be full size and capable of being put into production should the experimental work turn out favourably.

The work has been in progress for about three years. During that period approximately thirty tons of brass

* Emery Brothers Ltd., Birmingham, 6.

has been cast under carefully controlled conditions, much of which has been rolled to finished strip and sold to customers in satisfaction of ordinary orders or, where specially asked for, as "spill-free, superfine finish" metal. It has been definitely established that a superior quality of rolled brass strip can be produced from an ingot cast centrifugally under properly controlled conditions, and this report contains a very condensed description of the apparatus and of the experimental progress which has led to the success of the venture. The work is still in hand and some indication of the further lines of experiment which will be necessary to place the process upon an economic basis are indicated in the concluding section of this report.

II. Description of the Plant

The first thing to be done was to design and build a centrifugal casting machine which, whilst incorporating as far as desirable, the ordinary mechanical features of such machines, should at the same time be capable of allowing the special metallurgical technique of brass casting to be carried out, with such modifications as might be found necessary. Further, the dimensions of the spinning mould would have to be determined by external factors, viz., (1) the amount of molten metal conveniently available per cast, (2) the size of breaking down rolls available, and (3) the best shape (i.e., ratio of thickness, diameter and height) of ring having regard to the subsequent operations to be carried out upon it. After due consideration of these factors, a mould which would cast a ring of diameter 2 ft. 2 in. \times 15 in. \times $\frac{5}{8}$ in. thick was decided upon, such a ring absorbing the contents of one 150 lb. melting crucible. The special technique of brass casting is largely a question of providing a suitable reducing (or at any rate non-oxidising) atmosphere through which the molten brass is poured into the mould. This was accomplished (and herein lies one of the unique features of this machine and process) by the provision and maintenance of a large flush of coal gas in the spinning mould during the pouring operation. This necessitated a notable innovation in the design of centrifugal casting machines, viz., the transport of the *whole* of the supply of molten brass into the spinner at once, instead of (as in some machines for centrifuging iron alloys) allowing it to travel into the spinner from an outside container by means of a launder.

It soon became apparent that there would be a large number of variable conditions in the casting technique, such as (1) speed of rotation of mould, (2) mould material, (3) mould temperature, (4) speed of pouring, (5) temperature of molten metal, (6) duration of spin, (7) quantity and pressure of gas supply, (8) profile of pouring "lip," etc., etc., and since it was expected that a sound and satisfactory casting would only result from one combination of these variables, the method of experiment was to attempt to find that combination by a process of trial and error. Hence the machine must be as elastically designed as possible.

Notwithstanding this, a number of structural or other alterations was found to be necessary from time to time, some of which took several weeks to carry out.

Messrs. Stokes Castings Limited, an associated Company of the Sheepbridge Stokes Centrifugal Castings Company Limited, of Chesterfield, undertook to build a machine on the above-described lines. An existing

casting shop at Emery Brothers Works was adapted for the experiments by fitting it out with (1) the necessary melting furnaces, (2) a gas booster, (3) pyrometric equipment, (4) the necessary driving gear, (5) an auxiliary motor for "slow motion," (6) appliances for lining and firing the refractory of the "pouler box," (7) tackle, cranes, etc. for handling the machinery and the castings, which eventually weighed as much as 490-500 lb. each. Compressed air, pressure spray water, cooling water and other minor appliances were added later.

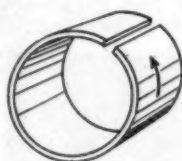


Fig. 1



Fig. 2

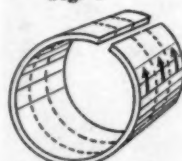


Fig. 3

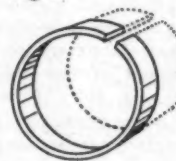


Fig. 4



Fig. 5



Fig. 6

The essential features of the machine will be seen in Fig. 7. A very substantial *Headstock A* is mounted on a shaft, carried in bearings on a heavy cast-iron bed *B* and is capable of being rotated at the speed desired by means of a belt drive through stepped pulleys *CCC*. Power for driving was supplied by a 20 h.p. motor, *via* a fast and loose pulley and a fluid flywheel. The latter was found necessary owing to the inertia of the headstock and the small power of the driving motor. The headstock *A* contained a removable cast-iron mould *D*, provision being made by means of removable flanges *EE* for (1) keeping the mould in position whilst in service, and (2) retaining the metal in the mould until it had solidified. The headstock shaft is hollowed out and a stream of cold water is circulated through it to prevent overheating of the main bearings by conduction of heat from the headstock.

A *Pourer carriage F* is provided with a pair of heavy bearings *GG*, which carry the *Pourer shaft H*. At the left end of the pourer shaft is mounted the *Pourer box*, described below. The pourer shaft is hollow; a supply of town gas, boosted up to a maximum pressure of 6 in. of mercury, is delivered *via* a flexible armoured hose *K* through the shaft to the *Pourer horns L*, situated within the pourer box.

A large handwheel, keyed to the other end of the

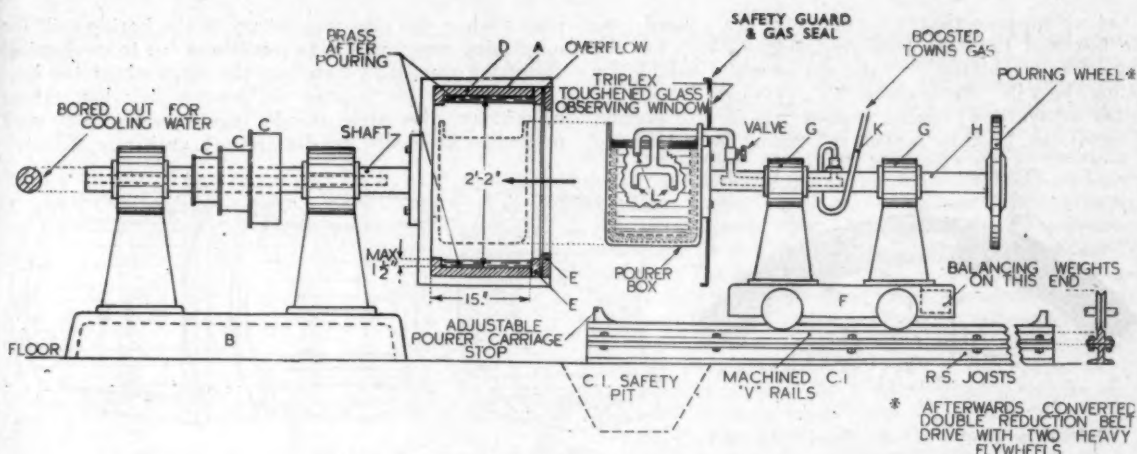


Fig. 7.—Essential features of the machine.

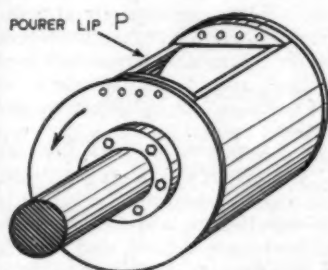


Fig. 8.—The pourer box. Note.—Pourer horns are not shown (see Fig. 7.)

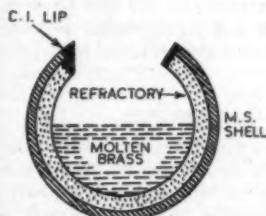


Fig. 9.—Section of pourer box in upright position.

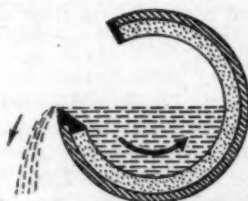


Fig. 10.—Section showing position of pourer box at start of pour.

pourer shaft, enables the pouring operation to be carried out by a man standing behind the pourer carriage. The whole of the pourer carriage can be pushed forward on rails until the pourer box goes completely inside the mould. The clearance between the two is a safe minimum. The axes of rotation of the pourer shaft and of the headstock shaft are in a straight line. Possibly the most important feature of the whole machine is the pourer box, which is shown in Figs. 8, 9 and 10. It is made of steel plate and lined by ramming a suitable refractory and fired. If the pourer box be partially filled with molten metal and rotated, it will empty itself over a *pourer lip* P, weir fashion, into the spinner. The length of the pourer lip, in relation to the 15 in. dimension of the mould, was a most important variable; and the contour of the lip and the metal of which it was made, were the subject of prolonged experiment during this research, in order that (1) a thin steady uniform "stream" of metal could be delivered over its edge into the spinner, and (2) the lip would stand up to the

action of molten brass without cracking or warping and without contaminating it. No less than seven shapes of lip and three different kinds of cast iron were tried before satisfaction was obtained, and there is still room for improvement in this direction.

III. The Casting Process

Before a cast can be made, it is necessary to heat up the pourer box to prevent chilling of the metal poured into it; and it is also necessary to preheat the mould. Sufficient heat is retained in the mould after the first cast to maintain it at a suitable temperature. Having made ready the required quantity of molten brass (either one, two or three pots of 160 lb. each), the headstock is set spinning to the speed decided upon for the day (between 250 and 550 revolutions per minute). The molten brass is then poured out of the crucibles in which it is melted into the pourer box, the latter being in the back position, i.e., to the right. The gas supply is then turned on and the pourer carriage pushed home so that, as previously described, the pourer box is entirely surrounded by the spinning mould. When it is considered that the space within is heavily saturated with coal gas, the metal is poured, by steadily rotating the handwheel (see Fig. 10) until the whole or nearly the whole of the metal is poured. It is thus caught up in the mould and flung by centrifugal force against the walls and in a few seconds has solidified. The pourer box is then withdrawn, the gas turned off, and the machine allowed to "free-wheel" for a pre-determined period, either until it comes naturally to rest, or until brought to rest by a foot brake. The ring will have shrunk somewhat, thus permitting of its easy withdrawal by means of a special device. It is then removed and allowed to cool naturally and sent to the machine shop after being weighed.

The rate of casting is governed partly by the melting capacity of the shop (six pit fires were available) and partly by the time taken in "setting up" and "knocking down." Also an ideal rate would be such that the inflow of heat into the mould per cast would be equal to the loss between casts, thus maintaining it at a constant temperature. When working on three-pot rings—weighing 490–500 lb. each—a good day's work was eight castings, i.e., about 35 cwt. Ideally, two shifts per day would be more economical or even a twenty-four hour run per day.

Let us suppose that x lb. of metal are poured into the box and that the cast ring weighs y lb. Let the ring, after machining, i.e., the net weight available for rolling, be z lb. Some metal, i.e., $(x-y)$ lb. is lost (by splash or overflow) during casting. A further portion of metal, i.e., $(y-z)$ lb. is removed during the machining

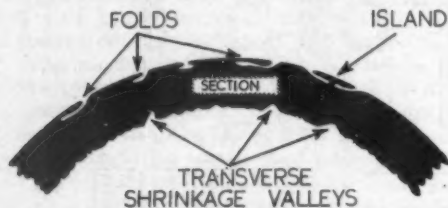


Fig. 11

Defects in shape in Figs. 12, 13, 14, are exaggerated.

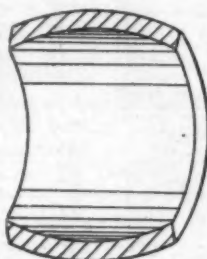


Fig. 12

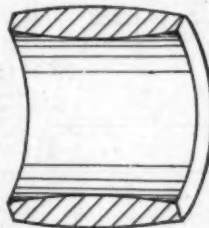


Fig. 13

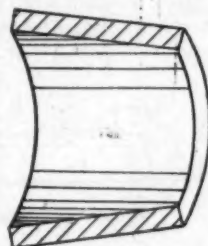


Fig. 14

process. The important thing to aim at is that the sum of the two losses shall be as small as possible; that is, casting and machining losses must be considered together. It might happen, for instance, that an extra six pounds loss permitted in casting would prevent a loss of, say, twenty pounds on the machining operation. This circumstance led to a long series of experiments of casting under different conditions, and although no finality has as yet been reached, machined castings weighing only some 15% less than the as-cast weight could be produced regularly, although occasionally one having a loss of 20% was made. All swarf and casting splash can, of course, be re-melted.

IV. Examination of the Castings

Below are stated the considerations which determined the character and extent of the examination of the cast rings:—

- (1) To decide to what extent the casting was sound, both in the surface regions and throughout its thickness.
- (2) To decide to what extent the rings lacked symmetry, i.e., whether thicker at the edges or in the centre, or whether they were not true cylinders or were suffering in any way from defects in shape which would cause an abnormally heavy waste on machining.
- (3) To learn something of the intrinsic properties of the rings as a cast product as compared with those of an ordinary chill casting of equal composition.

From the point of view of casting cylinders for rolling, the first two of the above are of immediate importance and the modifications in casting technique which were decided upon for any one series of trials were determined by the results of such examination. A good deal of information could be obtained from surface examination of the rings by eye or by means of a hand lens. In many cases rings were sectioned right through in three planes at right angles and polished in order to ascertain their degree of soundness and their variation in thickness. In other cases, macro-examination of whole sections was carried out by the usual polishing and etching methods. Reference to Figs. 11 to 14 will indicate some of the defects in the earlier rings, which were all overcome by varying the casting technique. Lack of true cylindricity, easily discoverable by presence of "high" and "low"

places when the ring was set up in the boring mill for machining, was found to be sometimes due to mechanical distortion caused by handling the rings whilst too hot.

As regards (3) it was not known to what extent superiority over strip mould ingots as regards such properties as density, tensile strength, grain size, etc., etc.,

would persist after the first breaking down and annealing, but it was thought that "better castings would make better strip." Apart from this, however, the numerical values of some of the above properties were determined mainly for their interest, i.e., considered exclusively as properties of spun brass castings. It is, therefore, not necessary to reproduce them here.

After several series of casts had been made, between which several structural alterations to the machine were carried out, a technique was evolved which resulted in ability to cast rings of excellent quality every time, weighing about five hundred pounds each, and capable of being machined for rolling with little overall loss. This technique has been standardised for the present and can now be carried out as a routine works' process by the ordinary casters without supervision from the author.

V. Machining the Castings

As this was considered to be a problem for the machine-tool engineer, enquiries were circulated in the trade for a machine-tool which would do the job quickly, cheaply and on a mass production basis. The author was surprised to find, amongst those who replied to the enquiry an amazing inability or disinclination to attack the problem by any other than strictly conventional lines. Estimates for time required and the cost of machining varied to an almost absurd degree. In the end, a temporary method was devised by the author and was made successful by the help of a Midlands' firm who supplied a vertical boring mill. This device is not ideal, for it is long, expensive and ill-adapted to a repetition job. It did, however, enable the work to be proceeded with and experience to be gained which would be of value in the design of a suitable one-purpose machine-tool.

Refer to Fig. 15.—Upon the table of the boring mill was fixed a circular cast iron attachment with raised bosses AA at intervals on the circumference of a circle of suitable radius. The casting was placed upon these as at BB. It was held very firmly in position by three steel forgings and three pieces of hard wood placed as at C and D, and tightened by the nut E. A "Wimet" (Tungsten Carbide) tool, provided with two cutting edges F and G, was mounted upon a vertical tool post, and the cutting edge F traversed across the entire vertical face. A radius tool on another post on the

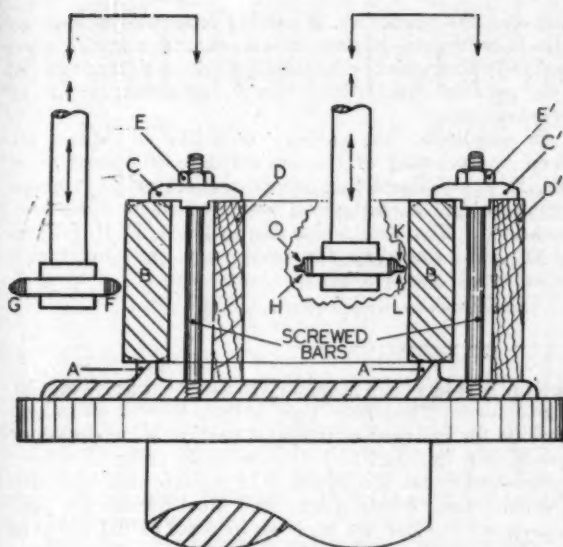


Fig. 15.—Machining the castings.

When clamps are inside the ring as at C D E, tool F does the outside facing, in the position shown, and tools O and H the outside radiussing, the tool post being outside and over to the right. When clamps are outside as at C' D' E', tool G does the inside facing, the tool post being inside the ring, and tools K and L do the inside radiussing, the R.H. tool post being inside the ring, as shown.

other side of the machine was brought into action so as to radius the top and bottom outside edges by means of the cutters O and H. The three supports and clamps and nuts C D E were then changed over to corresponding positions on the outside of the ring, as at C' D' E' and, by means of the cutting edge G, and the radiussing cutters K and L, the inside surface was machined and the top and bottom edges radiussed on the inside. Thus the left tool did all the facing and the right tool all the radiussing, each tool taking up positions outside and inside the ring as necessary. No changing of tools was involved. The whole operation took about an hour and a quarter, but further improvements and possible adoption of an entirely new method, as already mentioned, could be expected to lessen the cost and time of machining. It was found that if the rings were not radiussed they tended to crack out along the edges during the earlier stages of rolling—a defect which brings its own troubles in the mill at later stages.

After machining the rings, they were sawn through in two places at opposite ends of a diameter (see Fig. 5) by means of a metal-cutting band-saw, supplied by a local firm after being modified by them, in consultation with the author, to suit this work. In order to avoid cutting the rings in half and thus halving the maximum length of finished strip obtainable in one piece, a method of opening the rings after one saw cut is being considered so that they can be introduced full weight into the breaking down mill, as shown in Fig. 6.

VI. Rolling the Castings

A close observation was kept upon the metal during all the rolling, annealing, pickling, etc. operations involved in the production of the finished strip. No departure from ordinary mill practice was made. There was no trouble due to "ghosts" of machining marks,

which disappeared completely after the second rolling. Many of the strips rolled from some of the first experimental castings were defective, but several were saved from rejection by gouging out the defects with a portable milling tool. In the later stages of the work, providing the machining was done properly, an encouragingly high yield of good metal free from all spill and having excellent surface finish was obtained. Further experiment is in hand upon the maximum safe percentage reduction in rolling between anneals, particularly in regard to the first three rolling stages, and it is hoped that, owing to the enhanced toughness of the cast metal, as shown by bend and by elongation tests, this may be increased as compared with normal practice, thus possibly saving one or more annealing operation. It was also found that the "screws didn't require to be tightened down so much when rolling this metal" thus leading to a possible appreciable saving in power. A few castings were opened out in one piece and rolled as such. This presented no difficulty apart from that of man-handling such heavy weights. One whole casting was machined with a parting tool into a "helix"; each spiral being about 2 in. wide. This was equivalent to a flat strip $1\frac{1}{4}$ in. thick, 2 in. wide and some 45 ft. long as compared with the normal 3 ft. This helix was actually uncoiled and rolled in one piece down to 0.020 in. and then had a length of 940 yards!

VII. Tests on Finished Strip. The Future

It was considered that the reports of certain customers upon the behaviour of this metal during cold-pressing, stamping or spinning operations would be a more searching test of its quality than a series of conventional laboratory mechanical tests. At any rate, the former would furnish a more convincing argument when it came to selling the brass to makers of "Stamped Brassfoundry." Laboratory tests would afford useful confirmatory evidence, but to be convincing they must be carried out side by side with tests on ordinary brass strip of identical composition which has had *exactly* the same processing treatment from start to finish. So far there has been no chance of carrying out this set of tests. The customers who were most interested in the product were those who had suffered great inconvenience and loss by reason of defect in the metal not showing up until the article which was being made from it was nearly finished, e.g., dipped or chromium plated. One customer required brass sheet rectangles which after polishing, did not show up the *minutest* surface flaw. His report was that, whereas he had rejected as many as 45% of the rectangles made from ordinary brass which had looked good before polishing, the amount was reduced to 6% when centrifugal metal was used. Another customer, who makes very deep pressings which stretch out even the finest spill into deplorably bad markings, has specified spun cast metal and pays more for it. A third customer who usually purchases deep drawing quality brass (i.e., 66:34) finds that centrifugally cast metal in the cheaper grade (63:37) will draw equally well for his requirements.

At present, no publicity of the merits of this metal has been attempted for the reason that large supplies are not available, but the above instances will serve as an indication of its reception by customers. The quality of the metal has definitely been established, and were there no such thing as economics to be considered, chill strip casting might soon become a thing of the past as

far as brass strip is concerned. Nothing final can yet be said about the production costs of the process; there are obviously many modifications and improvements which have yet to be introduced. The present indications are, however, that despite the extra cost incurred by machining, the savings and advantages in other directions, some of which were listed at the beginning of this Report, coupled with a slightly increased price from certain customers, as already instanced, might ultimately lead to a regular routine production from a "battery" of machines fed from a large single electric melting unit. The sale of spun-cast ingots to the trade, for rolling in their own mills,

and also the possibility of casting other alloys, such as phosphor-bronze, gilding metals, cartridge metal, etc., and the melting and reclamation of our own scrap copper into rollable ingots have been visualised as future developments.

In conclusion, the author would like to express his deep appreciation of the enthusiastic co-operation of Mr. E. L. Williams,* at whose request and by whose initiative the investigation was embarked upon and sustained. Thanks are also due to Dr. J. E. Hurst and to Mr. W. Spedding for their very thoughtful and enlightened co-operation in many ways.

* Managing Director, Emery Brothers Ltd.

Co-operation Essential in the Iron and Steel Industry

THE British iron and steel industry has been discussed in the House of Commons and it seems that some modification has been made of the Government's original intention to take over the industry as a whole. The Minister of Supply has now set up a Steel Control Board which has clearly defined duties and do not include advice to the Government on how to carry out a policy of public ownership. This arrangement has clarified the situation and provides a solution to the difficulties which had arisen as a result of the Government's original announcement.

The iron and steel industry shows no inclination to be concerned with politics, but it is concerned with the problems of raw materials, production and distribution in this industry which are so vital to the prosperity of the nation. It is unfortunate, as the Rt. Hon. The Viscount Greenwood stated, in his address at the recent annual general meeting of Dorman, Long & Co. Ltd., that uncertainties during the past year have prevented as rapid progress as had been hoped with the major schemes of development included in the industry's five-year plan. This is true of the Dorman, Long project for a new steelworks and universal beam mill. A certain amount of preliminary work has been done and plans have been advanced, but greater progress might have been made if uncertainty regarding future ownership had not arisen.

Continuing, Viscount Greenwood said that shortages of fuel have a serious effect upon output and inadequate supplies of coal are immediately reflected in reduced production. Any failure to ensure the coal necessary to sustain operations at the coke ovens and furnaces means a fall in steel production and spells of enforced idleness for men employed on the plants. It is therefore, essential, when the collieries are taken over, that the Minister should not fail to ensure to the industry prompt and adequate deliveries of the fuel without which it cannot continue to exist.

Over a considerable period of years the Company has built up a large specialist technical staff for the purpose of promoting fuel economy. It has involved no small expenditure, but it has given valuable results in reduced coal consumption and greater economy in the use of all kinds of fuel. This field of activity is under constant review and we look for still further progress which will be of national benefit as well as of advantage to the Company.

Another factor affecting output is the acute shortage of railway locomotives and rolling stock. Because the railways have been unable to move finished products

promptly it has been necessary to carry excessive stocks. Customers at times have been obliged to wait quite long periods for delivery of material and on some occasions production has had to be curtailed in order to relieve congestion in the stockyards. In spite of this handicap Dorman, Long's output of rolled steel during the year amounted to 1,086,884 tons, an increase of 101,127 tons over the previous year, and it would have been larger had the firm been able to despatch promptly the products of the mills.

Referring to plant developments, Viscount Greenwood mentioned that although it has not been possible to proceed as rapidly as we had intended with the universal beam mill project, a considerable amount of development work has been undertaken. A new boiler house is nearing completion at the Cleveland Works. This plant, which will cost over £500,000 is expected to come into operation shortly. Also at the Cleveland Works a start has been made with the construction of a central ore unloading and ore preparation plant which will serve all the iron and steel works of the group. Important extensions have been carried out at the Bridge and Constructional Works where the area of the main engineering shops is being increased by 110,000 sq. ft. This development will enable the company to handle larger and heavier constructional steel units than have yet been fabricated in this country. The capacity of these shops will be increased by about 20%. Various other development projects are also in hand, together with the design and preparatory work in connection with the new steel works and universal beam mill, involve a vast amount of engineering activity, and the Board have set up a Plant Construction Department to undertake this work. The Central Engineering Department will continue to ensure the maximum production efficiency which is vital to the success of our programme and both departments will be under the control of Mr. E. T. Judge who has been appointed chief engineer to the Company.

Dr. L. B. Pfeil Honoured

It is noteworthy that Dr. L. B. Pfeil, Manager of the Development and Research Department of the Mond Nickel Co. Ltd., has been awarded the O.B.E. Dr. Pfeil has been responsible for many contributions to the advancement of metallurgical knowledge. During the war, he played an important part in the development of alloys for high-temperature service in gas turbines for jet-propelled aircraft, as well as for vital and secret applications of metals for service uses. His many friends will appreciate that he is a worthy recipient of this award.

Production of Magnesium Sand Castings

By G. B. Partridge

The design of a modern magnesium foundry is briefly discussed in which sections are partitioned off to effect good working conditions and also to facilitate production. Intelligent sand control is a necessary requirement and attention is directed to the methods applied and to the preparation of moulding sand for magnesium alloy castings. The preparation of moulds is considered, references made to the use of chills, to ingates and runners, and to risers, with a view to the production of sound castings.

THE late Mr. P. Pritchard foresaw some of the trials and difficulties of our modern existence and insisted that the foundry trade generally must improve its working conditions in order to function economically and attract sufficient numbers of the right type of worker. He used a phrase very much to the point when he said: "We have got to stop our foundries looking like coal mines."

The pre-war magnesium foundry in particular needed revision not because it was ill-lighted, cramped, or dirty, but because of the peculiar atmosphere created by the burning out of inhibitors in the moulding and core sands. In spite of many attempts at increased ventilation in the orthodox foundry building, as pouring took place adjacent to the moulder and knocking out was also done in the same bay, everyone working in it shared the foul atmosphere. When the demand for magnesium castings became so acute that foundry extension was essential, the opportunity was taken, in designing the new foundries, to try to overcome, in some measure, at least, this vexed problem of bad atmosphere.

Design of Modern Buildings

The latest type of foundry is illustrated in Fig. 1, and it will be seen that the basic principle involved is the partitioning off of the moulding and fettling sections from the metal melting and casting sections.

The area known as the core shop takes up almost the whole width of the bay at one end and contains the sand storage bins, sand dryer, mills and mixers to suit various types of sand mixtures, core stoves and sufficient working quantities of inhibitors, french chalk, etc., etc. Mono-rail conveyers take the tubs of sand to the mills and as cores are made on the various benches they are placed on a pendulum conveyer close at hand which takes them to the gas fired stoves for baking, after which they are stored on gently heated racks until required for use. The core stoves are of two types, batch and circulating, and are pyrometrically controlled as overheating of the cores causes loss of inhibitor, which increases the risk of scrap when subsequently used in the foundry.

A gangway separates the core shop from the moulding section where moulds are made in two distinct locations depending upon the size of job produced. It will be seen on referring to Fig. 1 that two endless moving tracks pass through the moulding shop, arrows indicating their direction of travel. The smaller one carries completed moulds up to box sizes 22 in. \times 18 in., whilst the other track accommodates large work, and boxes measuring 4 ft. 6 in. \times 2 ft. 6 in., containing crank case castings for heavy diesel engines are regularly conveyed on it. Moulders work each side of the track and their boxes are

fed on to it by ordinary roller track in the case of small and medium size work, heavy work being picked up by overhead crane and placed on the pallets bodily.

All moulds are put on boards or other supports before being placed on the rollers to prevent collapse of sand or accidents when handling the molten metal. Cranes are installed over the moulding areas marked 1 and 2 and can be used in cases where the floor machines are incapable of handling the job. Two sand recovery plants are in operation, in which sand from the knock-out is magnetized, screened, aerated and suitably wet up, after which it is delivered by overhead belt to the moulding sections. Such sand is generally referred to as "backing or ramming-up sand," the sand which is put next to the pattern face and called "facing" being freshly milled in the core shop.

The completed moulds are conveyed through apertures in the dividing wall to the melting shop where they are cast, and after passing through the cooling tunnel are knocked out and passed to the fettling shop which occupies the remainder of the foundry building along with heat-treatment and inspection sections.

In such a highly mechanised foundry production is very high if proper regard is given to the design and plating of patterns and, in order to prevent hold-ups, it has frequently been necessary to restart the track after pouring before the metal has had time to set. Some trepidation was incurred when this procedure was first adopted, but many thousands of aircraft castings have been made under these conditions and when subjected to the usual rigorous testing have not failed through defects brought about by this cause. It is fortunate that there is not the wide range of alloys in a magnesium foundry such as occurs in aluminium foundries, for if such were the case it will be realised that positioning the sequence of moulds to coincide with a particular melt would be a real problem; however, with the chief furnaceman keeping good liaison with the moulders and directing each particular batch of moulds everything usually works quite smoothly.

The main inhibitor in Elektron foundry sands is elementary sulphur which forms sulphur dioxide when burned in air giving rise to pungent atmospheric conditions, and a glance at Fig. 1 once more will illustrate how the majority of workers have been removed from the worst conditions. Some superficial burning of sulphur does take place in the moulding shop when skin drying of the moulds takes place, but the amount is never high and the main concentration occurs in the casting and knocking-out shop. As such a place is part and parcel of the foundry it has to be endured and huge suction fans deal with the fumes as soon as they are produced.

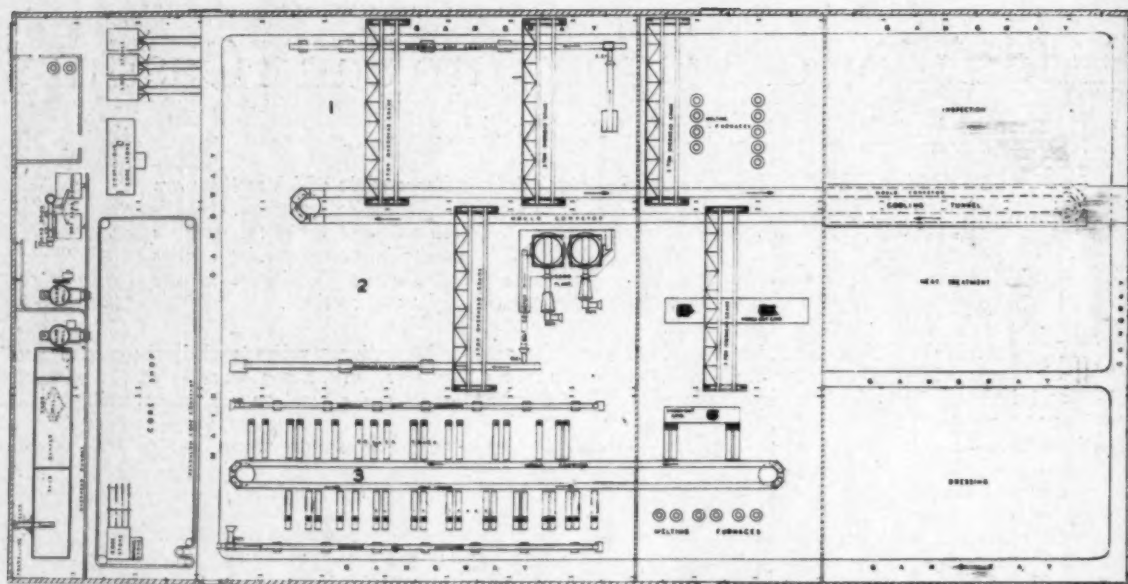


Fig. 1.—Drawing of a modern mechanised magnesium foundry.

Sand Preparation

The modern foundry and most of all the modern magnesium foundry cannot function efficiently and economically without some form of intelligent sand control to produce and maintain the right type of sand for the job. Let us be very clear on this point: It is not necessary for a laboratory ever to help run a foundry, for once man has found out how to make something he will continue to make it somehow and the mere fact that he may not have the services of a beautiful laboratory on tap is no deterrent. These two apparently divergent statements can only be reconciled by considering the actual facts of the matter, the first and most important being that the "job" has got to be made (often lost sight of by some metallurgists), the second, that it must be made well, and the third, that it must be made to compete, i.e., produced economically (these latter frequently overlooked by foundrymen). The facts of quality and economy demand the presence of a laboratory in a modern foundry and it must take control or the whole business house suffer inevitable loss. From time to time the work of the laboratory will be referred to but for the moment only in relation to its job of sand control.

Two types of sand are used for moulding magnesium castings: (a) Natural sand, and (b) Synthetic sand, and both are also made into cores although there are the other usual sands for core making which will be also dealt with.

(a) *Natural Sand.* The term natural sand is applied to material which is used for moulding purposes straight from the quarry with no other preparation except initial milling and it has been the basis of most foundries since their inception. It was not surprising therefore to find it being used for magnesium moulding when these alloys were introduced and magnesium castings have been made successfully for many years using such sand. Although one of the reasons put forward for its continuance is cheapness this is liable to be quite an error

Fig. 2.—Method of pouring a large casting on the track system (actual pouring just finishing).



Fig. 3.—Completed cast entering cooling tunnel.

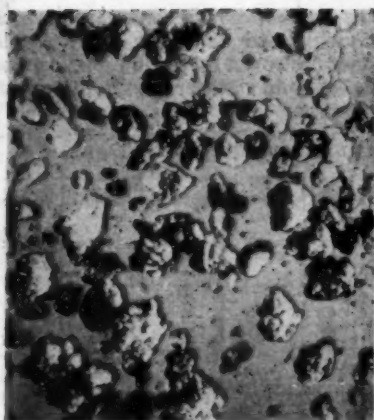


Fig. 4.—Bromsgrove sand, grains small size with clay adhering. ($\times 20$.)

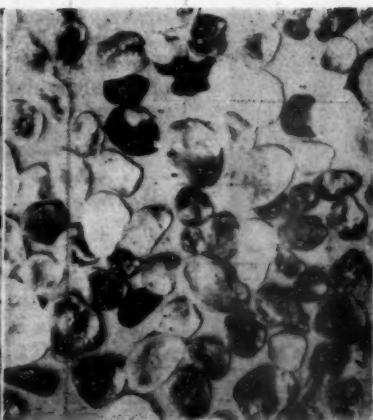


Fig. 5.—Congleton sand grains. A typical synthetic base. ($\times 20$.)



Fig. 6.—Leighton Buzzard 52. A larger type useful for excellent venting properties. ($\times 20$.)

as far as magnesium moulding is concerned, as will be shown later. Its advantages, however, are that on a cost per ton basis it is much cheaper than any other sand, because, apart from the fact that it is used straight from the sandpit, it is usually drawn from a local source, it gives a very good surface finish to castings due to its small grain size and it is usually a more comfortable sand for the moulder to work with, i.e., patching of a mould is a simple operation with natural sand. Fig. 4 gives an idea of its build-up which consists of more or less sub-angular silica grains coated somewhat irregularly with clay which is associated with red or brown iron oxide, also there is usually some organic matter present. As sands of the natural order contain relatively high amounts of clay, moisture contents of 5 to 6% are necessary before proper bond is developed for moulding operations.

On arrival at the works each load is sampled by the laboratory and then tipped into the storage bunker. The tests carried out on incoming loads are to determine moisture content, clay content, grading and microscopic examination of the grains, loss on ignition, and the results provide a valuable guide to the probable behaviour of the sand when in use.

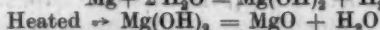
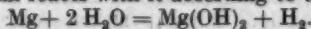
An actual example of control applied at this stage which did a large amount of good was when the laboratory first began to sample the incoming sand and found the clay content unnecessarily high. After discussion with the quarry owners, who said they could help by drawing from another seam, a striking reduction of about 3% clay occurred and for years afterwards the figure remained at the new lower level almost unwaveringly with consequent decreases of scrap in the foundry.

The general intake of sand into the foundry is by means of "facing sand" which consists of approximately two parts of natural floor sand and one part of new natural sand with the necessary inhibitors added, which in this case are sulphur and boric acid, and the whole well milled for 10 minutes. It is very important to insist upon a good milling time in order that thorough mixing shall take place and maximum bond developed. The quantity of inhibitors added may vary due to the size of section thickness encountered in a casting, but the nominal composition is 5% sulphur and 0.3% boric

acid which is adequate for normal castings. The new sand entering the foundry via the "facing" is not sufficient to maintain the strength of the bulk of the foundry sand which deteriorates due to burning of clay bond, although magnesium does not possess much heat content, and this is where the cost argument is liable to become unsettled for what is known as "virgin sand," i.e., new natural sand containing the necessary inhibitors must be added direct to the bulk sand from time to time.

Reference has been made to inhibitors and it is as well, at this stage, to attempt to explain their function. Magnesium and its alloys when heated are extremely reactive chemically towards air and moisture and this must be guarded against when making castings. Naturally, in conformity with the usual laws the reactivity, increases with rising temperature, but the way in which the molten metal is protected at high temperatures will be shown in the section on metal melting. Once the stream of molten metal leaves the casting ladle, however, it is a prey to rapid and sometimes violent oxidation which is prevented or inhibited by the use of these substances, sulphur and boric acid, but the volatile double fluorides are also used in some foundries. It must be made clear that the pouring stream is covered with a superficial layer of oxide which is good inasmuch that it acts as a tube through which the clean metal can pass, unfortunately, however, this skin is not continuous and easily breaks up with the consequent formation and accumulation of oxides and nitrides which can lodge in the castings as inclusions.

The use of sulphur is not clearly understood for it does not prevent burning once this has started but effectively prevents it starting if applied soon enough. It seems probable, however, that during its immediate conversion to SO_2 it absorbs the available oxygen thus surrounding the metal with a somewhat inert atmosphere, which, although still containing reactive nitrogen, appears to be satisfactory. Another problem, is encountered in the mould itself in the form of steam as magnesium reacts with it according to the equation:



As the mould contains the components of reaction at a high temperature, combination would proceed with



Fig. 7.—Laboratory control in the foundry. Taking moisture tests on the spot where it does most good.

extreme violence if not countered by the inhibitors of which sulphur dioxide gas is one. The boric acid is said to form a protective atmosphere and also some magnesium borate, but it certainly coats the sand grains, preventing silica reactions and protects the clay containing its combined water. The volatile salts surround the molten metal with a protective fluoride film or envelope which prevents reaction with the air and moisture.

Synthetic Sand. In referring to sand the term synthetic is used strictly in its correct sense which is "that which is built up from component parts" and it must never be thought that synthetic sand is a substitute and therefore inferior to the natural product. The United States of America is probably the true home of synthetic sand for they have available plenty of pure silica sand and major supplies of the now famous Bentonite clay which is quarried in Wyoming. Bentonite has remarkable bonding power which is mainly derived from its physical structure and as a foundry bonding material has yet to be equalled.

A washed pure silica sand is used as the base of synthetic sands (for typical example see Table 1 and Figs. 5 and 6) and the clay added along with the sulphur and boric acid at the initial milling. Water is added to obtain the required temper and it is usually the practice to also add a small quantity of ethylene glycol which has marked hygroscopic properties and prevents the sand drying out too rapidly, a usual fault with synthetic sands; some inhibitor properties are also ascribed to this chemical. The type of synthetic sand required can be obtained at will by varying the base sand, examples of which are shown in Table 1, and herein lies one of the chief advantages of this type of moulding sand: that its properties can be accurately predicted because it offers such complete control which natural sand can never do. Other advantages are: (a) the very good venting power or permeability which is usually many times that of natural sand; (b) the extremely low water content necessary to maintain adequate bond

strength, usually 2.5 to 3.5%, where natural sand requires 5.5% to mould with the same ease; (c) the Bentonite bond is not destroyed by the low heat content of the magnesium alloys and additions of virgin sand are rendered unnecessary.

TABLE I.—TYPICAL MAGNESIUM MOULDING SANDS.

Name	Broms-grove Red	Parishes	Congleton	Leighton Buzzard	Chelford
Type	Natural	Silica	Silica	Silica	Silica
Clay	8.0	—	—	—	—
Retained on 16	—	—	—	—	0.1
" " 22	—	—	—	2.0	0.4
" " 30	1.0	nil	1.5	4.5	3.0
" " 44	2.0	2.0	8.5	36.0	18.0
" " 60	3.5	19.5	27.5	39.0	41.0
" " 100	38.0	75.5	49.5	17.0	30.5
" " 150	28.5	2.5	12.0	1.5	4.5
" " 200	11.0	0.2	0.5	nil	1.6

As Bentonite possesses such high strength the quantity employed in the foundry sand is 3% (compare Broms-grove natural sand, Table 1) and this is the answer to the low moisture content of synthetic, the less clay or colloid the less water is necessary to spread its colloidal power.

There is another very real advantage when considering the difference in moisture contents of the two sands and that is the fact that when the synthetic sand moisture increases much over 3.5% it becomes impossible to work through excessive stickiness, yet natural sand can possess moisture contents up to 7 and even 8% and work quite easily in the foundry. The serious nature of this fact can be gauged truly when we realise that upon changing into steam, water expands to 1,600 times its own volume. During the war supplies of Bentonite were curtailed and an English clay named Fulbond was partly substituted, but although a good substitute and responsible for many thousands of moulds for aircraft castings, it did not equal Bentonite. A typical mix employed was 4% Fulbond No. 2 and 0.5% Bentonite.

Many thousands of good castings have been made using both natural and synthetic sand and to summarise the results of long experience, whereas in using synthetic sand it is possible to "get away" with faulty moulding technique, for example hard ramming, and have a low scrap return, in using natural sand the moulder, in addition to being a good one, should be a good technician, otherwise a large and mounting scrap pile is the result. It is unfortunate that the customer sometimes insists on a "glass bottle" finish, which, for an engineering casting, is frequently a mistake as it may be accompanied by a worthless inside.

The laboratory actually takes daily sand checks in the foundry, determining moisture content (Fig. 7), green compressive strength, dry compressive strength, permeability, green and dry, gas content, sulphur and boric acid determinations, and the foundry foremen are acquainted with the results as soon as possible, the information being useless if retained in the laboratory.

Core Sands. Core sands have already been referred to and many are made from the clay bonded sands discussed above. However, for intricate jacket work a sand possessing high flowability is necessary to "tuck" well in the core box, and for this type of work a proprietary compound of the molasses-lined oil type is preferred, plus inhibitors; its main drawbacks being the tendency to release quantities of gas when the metal is poured and being difficult to knock out through non-burning of bond. Careful control has to be exercised in the use

of such mixtures. If an oil sand core is required which by virtue of its shape needs to possess good green strength a proprietary mixture of the dextrine-linseed oil type is used, but recently the synthetic resins have received much publicity and no doubt will be extensively used for magnesium cores in the future if competitive enough in price.

Preparation of Mould

In a jobbing foundry many patterns have to be worked "loose" due to the small quantity of castings required, but when big orders or contracts are received for large or small castings, plating of patterns is an economic necessity, and if this is carried out skilfully potential output is considerably increased for two reasons: (a) the ease of working which automatically results from using a plated pattern and (b) moulding machines can be readily utilised.

There is nothing in magnesium moulding which prevents the use of the well-known types of moulding machine such as Adaptable, Macnab squeeze, Osborne jolt rammers and rollovers and these are regularly used for large scale production. The Sandslinger is also brought into considerable use and is of great value where correct planning of the layout has made use of all available space. It is advisable to use metal patterns in conjunction with the Sandslinger and a word of caution is needed regarding the moisture content of the sand, which must be carefully controlled for mechanical working if scrap is to be avoided. Providing that adequate green bond is maintained and renewal of inhibitors kept up in the sand it is unnecessary to prepare a facing sand, the foundry plant sand can be used for both facing and backing.

Theory and practice meet at the cross roads in no uncertain manner when the question of running magnesium castings arises, for although the ideal way of producing the thermal gradient necessary to make a sound casting is by means of top pouring, most magnesium castings are bottom poured. This method is adopted because the chemical activity of the metal induces oxidation and its light weight precludes any process which may induce turbulence. A compromise has to be sought, therefore, which fulfills the following conditions: (a) the formation of oxide skins and risk of moisture attack must be avoided; (b) the entry of metal into the mould must be smooth yet fast enough to fill the mould rapidly; (c) the risers must contain enough metal and therefore enough heat to invert the heat distribution from the ingate area to the top of the mould and so promote adequate feeding; and (d) solidification must proceed as rapidly as possible.

The result of attempting to apply these conditions in practice is first of all noticed with the downright or sprue, which is rectangular and usually measures approx. $1\frac{1}{2}$ in. \times $\frac{3}{4}$ in. Where a tubed hole through the sand is likely to produce a vortex and consequent oxidation the rectangular type discourages this formation and if the casting to be poured is large and deep, several of these rectangular downrights are used, each separated by about $\frac{3}{4}$ in. of sand; this arrangement prevents overheating of the sand passage.

The depth of the mould is taken care of by breaking the drop by means of a runner usually at a joint line thus reducing the tendency to splash and cause oxidation. The runners are of the type shown in Fig. 8 and are designed to catch dross and oxides in the narrow upper

portion so that only clean metal shall flow through the ingates (also shown) into the casting. They are frequently of the double type and act as extra safeguards in quietening the metal stream and preventing entrance of oxide into the mould. The relationship of sprue, runner and ingate, is decided for each particular job by the foundry, considerable information on which has been published.*

Fig. 8.—(a) System of running and gating a small casting.



Courtesy of Birmid Industries

Fig. 8.—(b) Small test casting with typical runners and risers.

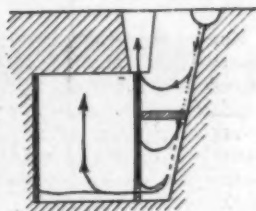
As magnesium alloys have, in general, a wide solidification range, low specific gravity and heat content, the provision of large risers to feed heavy sections is very necessary and, as previously mentioned, compensation has to be provided for the effects of bottom pouring. Care must be exercised in the design of risers, for it will be seen that if they are deep yet narrow, freezing of the neck may take place rendering the upper portion of the riser useless. Riser cavities are sometimes lined with an insulating material as an additional safeguard against this happening.

Cast iron chills are used to equalise the solidification rate of thick and thin sections located deep down in the casting and take the place of risers where it is impossible to apply them. The size and thickness of the chill depends upon the section thickness of the casting at any one point, but the attempt is always made to promote

* "Gating, Riser and Chilling of Magnesium Sand Castings." By A. Cristello. *The Foundry*, May, 1945.

directional solidification, overchilling is liable to cause defects elsewhere in the casting and should be avoided. All chills are lightly sprayed with a bonded talc or red oxide mixture to retard moisture condensation and large ones contain holes to prevent "blowing."

Other forms of running magnesium castings do exist the purpose in each case being to apply hot metal to the upper parts of the mould and finally to the riser, i.e.,



Courtesy of F. A. Hughes & Co., Ltd.
Fig. 8. (c) Slit gate diagram from "Technology of Magnesium and its Alloys."

the casting is run first at the bottom, then a middle system of ingates takes over and finishing with a system located into the riser head, the metal automatically entering each system as it finds its own level. One type of runner calling for special mention is called the "slit gate" and is useful in slowly poured thick walled castings in which the metal in the "slit" rises in conjunction with the metal in the casting. For this method to succeed pouring must be steady or the metal stream will persist in continuing to the base of the runner and mould instead of progressively rising as the mould is filled, and with this open type of runner oxidation will easily occur unless the protective atmosphere is very carefully maintained.

The Geology and Mineral Resources of Ceylon

By L. J. D. FERNANDO, M.Sc., A.R.C.S., D.I.C.

THE above subject was discussed in the sixth of a series of lectures organised by the Imperial Institute. The lecturer, Mr. Fernando, dealt with recent progress in geological investigations and mineral developments. Describing first the topography of Ceylon, he dealt in turn with the three well-defined and easily recognisable peneplains. The first terrace or coastal plain, ranging in height to about 400 ft. above sea level, reaches its maximum development in the northern half of the Island, and, with the exception of isolated buttes or monadnock-like hills, is devoid of relief. The second at an elevation of about 1,700 ft. rises steeply in most parts from the coastal plain although it is not easily recognisable from the north and east. Rising from the second terrace and forming the central highland country is the third terrace at a general altitude of about 4,000 ft. Here the highest mountains of the Island occur, of which Adams Peak (7,360 ft.) is the best known, although not the highest.

Nearly nine-tenths of the area of Ceylon is covered by rocks belonging to the Archaean, the oldest of the geological systems. They consist of highly metamorphosed sediments, provisionally grouped with the Khondalite Series of India, resting on an older group of granites, gneisses and schists, provisionally termed the Vijaya Series. The remaining area is covered by small occurrences of shallow water, non-marine deposits of Upper Gondwana age (Jurassic) in the neighbourhood of Tabbowa and hence named the Tabbowa Series; the Jaffna limestones of Miocene age mainly in the north-west of the Island; Pleistocene gravels overlying the gneisses of the coastal plain in the north west; and recent deposits including the residual and alluvial deposits of the Island, especially in the west.

Graphite mining is the largest and most important mineral industry in the Island, the deposits being the most important in the world. Mining of this mineral has been carried out for well over 100 years and for several decades Ceylon has been the world's principal source. The economic deposits are vein deposits which appear to be confined to the Khondalite System. The number of mines varies considerably from time to time, depending largely on the demand and market prices. At the peak production of 1941-42 there were well over a thousand mines, the great majority, however, being no more than

pits working at shallow depths from 60-150 ft. There are about six large mines, the best known of which are the Bogala mines, near Ruanwella, and the Ragedara and Kahatagaha mines in the Kurunegala district, the last having a depth of over 1,600 ft. Mechanical haulage is installed in these and the underground workings are electrically lit. The entire production, which reached a peak in 1941 (27,232 long tons) and 1942 (27,734 long tons), but has since fallen, being only 7,820 long tons in 1945, is exported. The reserves of the mineral appear to be unlimited.

Ceylon has long been renowned for its gems, the industry itself being of the greatest antiquity. With the possible exception of Brazil, no other country in the world produces such an abundance and variety of precious and semi-precious stones. The long list of gemstones won includes rubies, sapphires, chrysoberyl, beryl, topaz, zircon, tourmaline, spinel, garnet, moonstone, amethyst and many others. The gem-bearing area comprises between 600 and 800 square miles in the south-west sector of the Island and is centred round Ratnapura, "the city of gems." With the exception of moonstones and possibly a few tourmalines and garnets which are found *in situ*, the gemstones are all obtained from old alluvial deposits. The simple procedure for winning the gems and rather primitive gem-cutting methods were described by the lecturer. One serious fault of the local cutter is that he has a tendency to cut for weight rather than for appearance. Associated with the gems in the gem-bearing gravels or "illam" is an unusual assemblage of rare earth minerals. Unfortunately the heavy residues including these are normally discarded and many mineralogically interesting specimens are lost. At the present, however, none of them has any commercial importance. Workable deposits of one or more of these minerals may, however, be discovered as the result of future geological work.

Ilmenite, monazite and zircon are found in the easily accessible form of black sands along wide stretches of the coast, but there is no production at present. Other mineral deposits of potential economic importance include iron ores, kaolin, magnesite, glass sands and peat. There is some small production, mainly for local use, of kaolin and quartz sands. Many deposits of these minerals, however, remain unexploited or worked haphazardly in an indifferent way. Nearly all of them could form the basis of a valuable export trade or of a number of profitable local industries.

Modern Methods of Gas Analysis

Part I—Importance of Accurate Analysis, Types of Gases, and Collection of Samples

By W. D. Vint

Any raw material used in the manufacture of metals and its products should be accurately assessed with regard to its importance. Gas is no exception and it should be accurately analysed with the object of maintaining certain qualities

which are important in effecting economy in fuel, quality of product, and ease of control. In the present article—the first of a series—the author discusses the importance of accurate analyses, types of gases, and collection of samples.

IN a surprisingly large number of industrial research and analytical laboratories, the gas testing department is designated to a small corner of the main building, supplied with a meagre collection of obsolete apparatus and other odds and ends and expected to produce results comparable with gas referee's results.

The blame for the insignificance attached to the gas department is probably due to the fact that in comparison with the rest of the work, little gas analysis is required to be done. Another point—how many metallurgists are gas-minded? Take the simple case of the single blast furnace. Iron ore, limestone, coke, and pig-iron, etc., are very carefully analysed and tabulated probably at least once per shift to be proudly exhibited in a large ledger for the use of all and sundry. An enquiry for the analysis of the blast-furnace gas elicits the information that this analysis is very rarely made; blast-furnace gas is a waste product as far as pig-iron production is concerned and even if it is used for pre-heating stoves or installations requiring heat, its analysis is not considered important. The object of the works is to turn out pig-iron, not gas, and thus the short-sighted attitude continues to an obviously derogatory effect upon production.

Any raw or waste material whether solid, liquid or gas concerned in the manufacture of metals and their products should be accurately assessed with regard to its importance. Quite often, because gas is invisible and possesses little tangibility, it is regarded as something which is there but does not matter. In the larger metallurgical concerns, a separate department is allocated to gas testing and proper significance attached to that department. The smaller concerns are not

so fastidious; a chemist to analyse their products sold on specification is all that is needed. The necessity for constant and accurate gas analysis is at once evident if the following results are to be achieved.

(1) Economy of fuel. (A large item indeed when production costs are assessed.)

(2) Quality of product. (Good quality is impossible to obtain under fluctuating conditions actuated by variations in gaseous fuels.)

(3) Ease of control.

The last named is perhaps not so obvious, as it applies more to the heat-treatment side of the business. Controlling the temperature of a heat-treatment furnace is much easier if a

material analyses, and this is an impossible feat without the right equipment.

In subsequent issues, I propose to deal with the standard up-to-date gas analysis apparatus with a discussion upon their merits and demerits and their suitability for dealing with the analyses likely to be encountered on the works.

Types of Industrial Gases

In ferrous metallurgical industries gases may be divided into two main categories:—

(1) Gases used for heating.

The average chemical composition of some of these gases is given in the following table.

	Town Gas	Coke Oven Gas	Producer Gas	Blast Furnace Gas	Blue Water Gas	Carburetted Water Gas	Oil Gas
CO ₂ ..	5%	3%	7%	10%	4.9%	5.5%	—
O ₂ ..	0.3%	0.3%	0.1%	Nil	Nil	0.4%	—
CnHm ..	3%	3%	0.7%	Nil	Nil	6%	35%
CO ..	13%	7%	23%	29%	40%	30%	—
H ₂ ..	23%	26%	3.5%	—	1%	15%	40%
CH ₄ ..	48%	56%	12%	2.5%	50%	37%	25%
N ₂ ..	—	—	—	—	—	—	—

Nitrogen percentages are obtained by difference from 100.

knowledge of the calorific value of the gas is known. Also flame characteristics and the location of hot spots, etc., can be estimated if the gaseous constituents and their percentages are known.

Not only should a good system of frequent gas testing be incorporated if the maximum efficiency is to be obtained from a plant, but great care should be exercised to ensure that accurate analyses are reported. This particular section of the laboratory work should not be considered as a spare-time job, something to be hurried over by one of the lesser skilled chemists, reported, and henceforth forgotten, but a job warranting a deal of time, patience, and skill. There is little doubt that correct gas analysis requires more skill than most other

(2) Gases used for bright annealing atmospheres.

A large number of different types of atmosphere gases for bright annealing are appearing upon the market and their compositions are equally numerous and varied. They possess one common characteristic, viz., total absence of free oxygen. They are made up of varying percentages (usually small) of CO₂, CO, H₂, with the remaining diluting constituent of nitrogen. From the point of view of the effect different ratios of these gases have upon the surface of steel, accurate analyses must be made.

From the foregoing, one may observe that, with few exceptions, practically all the gases likely to be encountered consist of no more than seven separate constituents and an apparatus designed

for estimating these constituents should have a universal use. Unfortunately, however, the more common piece of apparatus possessing a tolerable accuracy of to within 0.1% and capable of dealing with these seven gases is quite useless for such jobs as require an accuracy of to within say 0.01%. Correct analyses of atmosphere gases is particularly important especially when a trace of oxygen can impair the bright surface of metal products.

On the other hand, a delicate apparatus capable of analysing constituent gases to within 0.01% is unnecessary with the normal run of heating gases where a discrepancy of say 0.1% is not serious.

The types of gas analysis apparatus now on the market are becoming almost legion and although they all possess their own points, a sharp discrimination should be made when purchase is intended. The following considerations should primarily be taken into account.

(1) Purpose for which the apparatus is intended.

(2) Accuracy required.

(3) Degree of skilled labour available for its operation.

There are very few pieces of apparatus on the market possessing really original design as most are nearly all modifications and improvements on older standard designs.

Methods of Estimating the Common Constituents of Gases

Carbon Dioxide.—Absorption by strong alkaline solutions such as caustic soda or potash is the method universally used. The acidical properties of CO_2 and the strong alkalinity of the caustics render an almost instantaneous reaction. It should be noted that other gases are soluble in NaOH and KOH solutions, viz., chlorine, hydrochloric acid gas, cyanogen, hydrogen sulphide, and sulphur dioxide. Should any of these gases be present in the sample to be analysed they should be previously removed before the absorption of CO_2 .

Oxygen.—Pyrogallol made alkaline with caustic soda or potash is here again to be recommended for its ease of handling and undoubted reliability. Various other reagents have been used from time to time such as sodium sulphite solution, yellow phosphorus, metallic copper, chromous chloride (chromous chloride is particularly suitable for oxygen absorption should such gases as H_2S or CO_2 be present and

which have not previously been removed). It can, however, be safely stated that for general purposes, alkaline pyrogallol is the most satisfactory reagent.

Unsaturated Hydrocarbons.—A variety of reagents have been used for the estimation of the unsaturated hydrocarbons, viz., bromine water, fuming sulphuric acid, etc., but in the opinion of the author, bromine water has proved the most satisfactory. Fractional burning is a comparatively new method which will be discussed in a later article.

Carbon Monoxide.—A choice of several reagents here again exists, but acidical or ammoniacal cuprous chloride solutions are generally adopted and have several redeeming features. Acidical cuprous chloride is particularly recommended on account of its capacity for regeneration by means of metallic copper.

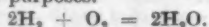
Carbon monoxide can be estimated by means of explosion with oxygen or air.



Thus two volumes of carbon monoxide produce two volumes of CO_2 which can be estimated by means of absorption in caustic soda. An industrial gas containing CO , however, usually contains other combustible gases such as CH_4 and H_2 , these giving rise to complications in the volumes of gases produced after explosion, therefore it is much more usual to remove the CO beforehand by absorption in cuprous chloride solution. No oxygen should be present in the gas before estimation of the CO ; oxygen and carbon monoxide alike are absorbed by the cuprous chloride.

Percentages of 0.5 and above are quite satisfactorily dealt with by means of acidical or ammoniacal cuprous chloride solution but for smaller amounts, a special type of apparatus is required. This method depends upon the fact that when carbon monoxide is passed through a heated tube containing iodine pentoxide, an equivalent amount of free iodine is released which, when absorbed in potassium iodide solution can be titrated with a standard sodium thiosulphate solution. A more detailed description of this method will be dealt with in a subsequent article.

Hydrogen.—Percentages of hydrogen in heating gases are generally high enough to be estimated by direct explosion with air or oxygen and the method is sufficiently reliable for all normal purposes.

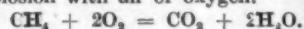


That is, $2/3 \times$ the contraction is equivalent to the hydrogen originally present. The volume of condensed water is quite small enough to be neglected.

For more accurate purposes the hydrogen is best estimated by passing over heated paladinised asbestos. The slight error incurred by exploding hydrogen with oxygen is here eliminated, but a reversion of the heated gas to room temperature must be assured, otherwise a much larger error can be introduced.

Other reagents which can be used for the absorption of hydrogen are Paladium Black (a finely divided form of metallic paladium) or molten sodium or potassium metal at a temperature between $300/400^\circ\text{C}$.

Methane.—The best method of estimating this constituent is by explosion with air or oxygen.



That is, one volume of methane produces one volume of carbon dioxide which can be easily estimated by absorption in caustic soda.

Nitrogen.—A constituent of most industrial gases, this gas is obtained by calculation.

$$\% \text{ nitrogen} = 100 - (\% \text{CO}_2 + \% \text{O}_2 + \% \text{CO} + \% \text{CH}_4, \text{ etc.})$$

The main constituents of heating and atmosphere gases can thus be estimated with the ordinary forms of Orsat, Bone and Wheeler, or Hempel apparatus.

No heating or atmosphere gas, however, consists solely of the above constituents. There are varying degrees of impurities by such gases as SO_2 , H_2S , NH_3 , NO , etc., which are very often required to be known. Special methods, independent of the usual gas analysis apparatus have been devised to deal with these common impurities and will be dealt with in a subsequent article.

Waste Gases

Waste gases can hardly be said to fall into either categories of heating or atmosphere gases, but they are of paramount importance if an accurate heat balance is to be obtained. There is little need for accuracy greater than 0.1% and the ordinary portable Orsat is adequate.

Waste gas analyses should indicate the percentages of CO_2 , O_2 , CO , and N_2 . From these percentages, information regarding the efficiency with which the fuel is burnt can be gained. Most of the larger metallurgical concerns employ electrical CO_2 and CO recorders which plot a continuous

record throughout the whole 24 hours. This type of installation can be considered ideal. Even in these larger plants, the faithful Orsat is always at hand should a check on the electrical recorder be required. Smaller works are not in the fortunate position of being able to afford expensive electrical recorders for all their boilers and furnaces and spot tests with the Orsat are the best that can be managed.

Sampling Methods

Little doubt can be felt that correct gas sampling is an even more difficult and exacting operation than the sampling of any other liquid or solid material. The high diffusive properties of gases render adulteration by the surrounding air a very simple matter and their invisibility is an effective mask against such an adulteration. Fortunately, this high degree of diffusivity has an advantage in that it renders mixtures of gases extremely homogeneous. Leakage from the confining vessel or diffusion of air is a calamity which must be guarded against.

length of time that pumping should be continued to obtain a perfect sample of the gas. If a hand pump or bellows is not available, the gas can be collected by previously filling the tube with mercury, strong, gas saturated brine, or gas saturated acidulated water, and collecting the gas by means of displacement. The latter expedient has the slight disadvantage that unless mercury is used, small amounts of gases (especially CO_2), can be dissolved by the fluid with the result that slightly lower figures will be obtained. The error, however, is so small as to be completely neglected for all ordinary purposes. This latter method must only be used when the gas is under positive pressure.

A continuous sample over long periods can be collected by using the aspirator and connections as shown in Fig. 3. The slight vacuum produced by the escaping liquid must be more than counterbalanced by the gas supply pressure, otherwise air can be sucked in from the blow-off tube.

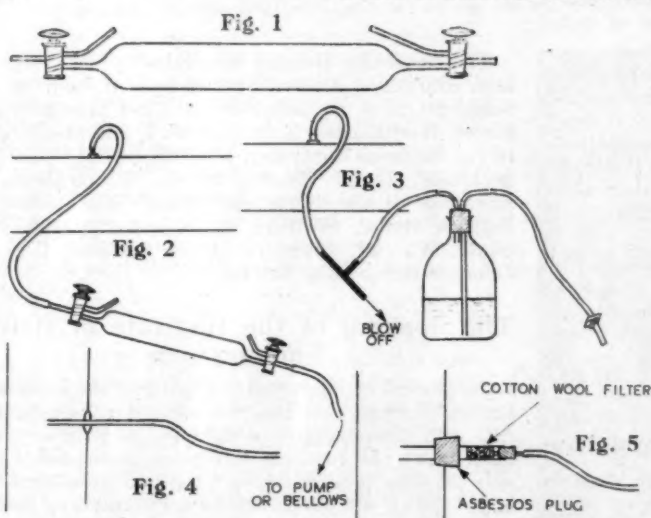


Fig. 1.—Gas sampling container. Fig. 2.—Arrangement for obtaining sample of gas. Fig. 3.—Showing the application of an aspirator. Fig. 4.—Inserting tube into gas stream. Fig. 5.—The use of a cotton wool filter.

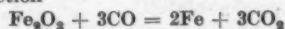
The most reliable gas sampling container is a glass tube of the pattern shown in Fig. 1. The taps must be well greased and the tube should be capable of holding a vacuum for several days. A hand pump or bellows should be connected to one side of the tube, the other side of the tube being connected to the gas sampling point. The set up is simply illustrated in Fig. 2. Experience will prove the

When dealing with water soluble gases, NH_3 , SO_2 , N_2O , etc., displacement methods by means of liquids should never be used; (mercury does not come into this category). A perfectly dry tube with use of the hand pump is the only method which can produce accurate sampling. Care must also be taken to ensure that the gas does not come into contact with water before it reaches the apparatus.

Waste Gas Sampling

The correct sampling of waste gases is not quite so easy as it seems. For reduced pressure whilst turbulence and air leaks through cracks in the brickwork can produce adulterated samples. The gas sampling point should be taken such that no long length of flue separates the point from the fire. The longer the length of flue the greater the possibility of air leaks impairing the accuracy of the gas sample.

A tube should be inserted as far into the centre of the gas stream as is possible and the point of ingress should be well lagged (see Fig. 4). The choice of tubes for sampling is important. If the inside flue temperature is below 250°C ., an iron tube can be used. Above this temperature, the reaction



proceeds with an obvious deteriorating effect upon the sample. Pyrex tubes can be used up to 600°C ., at which temperature, sagging is likely to take place. Above 600°C ., a quartz tube should be used.

Dirty gases (e.g., uncleaned producer gas), can be sampled in the usual manner of tube and pump but to prevent the capillaries of the taps becoming clogged, a filter of cotton wool should be inserted before the tube (see Fig. 5).

All-Aluminium Ships for the Alcoa Steamship Co.

FROM time to time reference is made to the application of aluminium and its alloys in shipbuilding, but apart from a few small vessels, in which aluminium is the principal constructional material, the use of this material has been limited to superstructures. It is noteworthy, therefore, that Mr. Harman Lewis, president of the Alcoa Steamship Co., has announced plans for the first all-aluminium ships for the company. Two vessels are contemplated, the larger of which will have a carrying capacity of 8,000 tons, 1,500 tons more than a steel ship of similar dimensions. It will be 400 ft. long and Diesel-driven. The small ship will be steam-powered. It has been estimated that the ships will be in service within eighteen months and two years after placing the contracts. It is expected that their successful completion will lead to extensive use of aluminium in shipbuilding.

Nickel in 1946

THE world's deliveries of Canadian nickel to firms in 1946 will compare favourably with those of 1945. This was stated by Mr. R. C. Stanley, Chairman and President of the International Nickel Company of Canada, Ltd., in a letter recently addressed to the shareholders, in which he surveyed the nickel industry during the last year.

The outstanding feature of his review is the rapidity with which the Company has been reconverted to post-war conditions, an achievement which owes much to long established and comprehensive research activities in this country, Canada and U.S.A.

Operations in Canada are now running at 75% of the enlarged productive capacity, which closely approximates the rate prevailing in 1937, when a record tonnage of nickel was sold. Although the costs of mining, smelting and refining have increased substantially, apart from bringing the U.S. prices of nickel into line with the prices in the world market, no price raise has been made, or is contemplated.

Trends of consumption during the past year show that over 35% of the nickel used in the world is absorbed in stainless steels, which are likely to find increasing markets, particularly for industrial equipment.

Plating accounted for more nickel in 1946 than in any previous year, showing an increase of over 50% on pre-war consumption, and, in U.S.A., there was a similar spectacular increase in the production of nickel silver.

There are many new, revived, or extended applications for nickel steels, nickel plate, cupro-nickel and the Company's well-known alloys, Monel and Inconel. These are now joined by the Nimonic series of alloys, originated and developed by The Mond Nickel Company, Ltd., which, having made jet-propulsion in this country a practical possibility, continue to make a major contribution to our supremacy in this sphere.

Mr. Stanley stresses the commercial economies which have resulted from new applications for nickel, and, with industry more than ever aware of the importance of such economies, he considers that the outlook indicates expanding sales in the world markets for nickel.

Design for Progress Award Programme

As in former years the James F. Lincoln Arc Welding Foundation is again sponsoring an award programme with the object of encouraging and stimulating scientific interest in and scientific study, research and education in respect of the development of arc-welding industries, through advances in the knowledge of design and practical application of the arc-welding process. On this occasion, it is termed, "A 200,000 dollar Design for Progress Award Programme," and provides for 452 awards for papers embracing every field of industry and research where arc welding may be applied with attendant benefits. There are certain rules and conditions to be observed in connection with the programme, but it is important to observe that papers presented should be in hand by June 1st, 1947. All communications relative to this programme should be addressed to the Secretary, James F. Lincoln Arc Welding Foundation, Cleveland 1, Ohio, U.S.A.

The Control of Iron and Steel (No. 55) Order

THE Minister of Supply has made the Control of Iron and Steel (No. 55) Order, which came into force on January 1, 1947. The Order amends the maximum prices of certain steel products, the principal alterations being increases in the price of wire ropes, brake cable, tinned steel wire, and high carbon cold-rolled strip; the Order reduces the quantity of conduit tubes that may be acquired without licence; and releases shell steel discards from price control. A detailed list of Related Schedules, Nos. 130 to 142 inclusive, is outlined below:—

Old No. Revoked	New Schedule No.	Product	Remarks
17	130	Blooms to Admiralty Spec.	Normalising extras on small sizes increased.
115	131	Alloy Steel Billets and Bars	Schedule re-edited, minor corrections.
26	132	Tinplate and Sheet Bars	Ditto
36	133	Cold-Rolled Strip	Revised extras and allowances on High Carbon Quality.
124	134	Mild Steel Wire and Wire Products	Tinned wire prices increased by from £1 to £6 per ton.
81	135	Stitching Wire	
84	136	Brake Cable	5% Increase.
126	137	Special Patented Wire	Tinned wire prices increased by from £1 to £6 per ton.
89	138	Patented Roping Wire	5% Increase.
90	139	Locked Coil Ropes	Ditto.
91	140	Wire Ropes	Small quantity extras clarified.
96	141	Railway Tyres and Axles	Minor amendment.
129	142	Merchants' Consolidated Price Schedule	
30	—	Military Bridge Sections	Withdrawn.

The remaining Related Schedules to the Control of Iron and Steel Orders are unchanged, and all the Schedules may be inspected at Steel House, Tothill Street, Westminster, London, S.W. 1, and at the offices of the Regional Controller, Ministry of Supply, C.M.L. Buildings, Great Charles Street, Birmingham, 3; Britannia House, Fountain Street, Manchester, 2; Fargate House, Sheffield, 1; Clayton Street, Newcastle, W.; 21, Glassford Street, Glasgow, C.1; 14, James Street South, Belfast.

The Journal of the Institute of British Foundrymen

READERS will be interested to learn that the Institute of British Foundrymen has commenced a new journal. Although comprising only eight pages, it is a venture upon which the Institute can be congratulated; probably as time proceeds it will tend to grow, but it is hoped that it will retain the form indicated by the first two numbers. It is dignified in presentation, and there is that personal note about it which will undoubtedly be attractive to members. We hope it will flourish.

Aluminium Development Association Move to Larger Premises

DUE to the increase in the activities of the Aluminium Development Association and the consequent expansion of the staff, it has been found necessary to move from 67, Brook Street, London, W.1, to more suitable premises.

From January 6th the headquarters of the Association will be at 33, Grosvenor Street, London, W.1. The telephone number remains unchanged—Mayfair 7501.

Some Archaic Mining Apparatus

By Douglas Rennie Hudson, B.Sc., Ph.D.

(University of Leeds)

"En résumé,—et c'est là un point d'une grande importance historique,—ces mines du Laurion faisaient d'Athènes le principal et presque unique marché d'argent et de plomb du monde grec, une Swansea ou une Londres de l'antiquité, si l'on nous permet cette comparaison."

DE LAUNAY.

"For the Phoenicians, it seems, were of old clever men at making discoveries for their own gain, and the Iticians equally adroit in leaving no gain to anybody else."

DIODORUS SICULUS,

Library of History, 5, Chap. 35, 37.

ABSTRACT.

In pre-Christian centuries Andalusia was a virgin field of great richness and extent, in copper, lead, silver, and gold; the silver-lead deposits of Laurion were probably worked even earlier. A synthesis is attempted for dewatering equipment. By comparison with ancient workings, the methods described by classical authors turn out to be unexpectedly accurate. Apparatus used consisted of the Archimedes' screw (*cochlea*) and the treadmill water-wheel driven by two slaves. Considered mechanically, these were not very efficient but, in comparison with the human chain, the ancients were delighted with their excellent performance. Relics, when recovered, compare very accurately with descriptions and specifications of Vitruvius in the first century. They can be correlated strictly with a contemporary drawing from Pompeii, and later illustrations from his manuscript works. Another type of water-wheel with endless chain, quite recently in use in Persia and Cyprus, carried across to the New World by *conquistadores*, was exhumed from a mine in Darien at the end of last century.

ACCUSTOMED, as we are, to extracting the last ounce of mine from a lode, and the last penny-weight of metal from this mine, it is not easy to realise what wealth of placer and gossan was available to the ancient metallurgists.

"In Paeonia, they say, when showers of rain fall continuously, as the soil melts away, gold is found called unfired (*ἀσφύρα*) . . . the ground is so full that many have found more than a *mina's* weight." ARISTOTLE, *On marvellous things heard*, 45. (1 *mina*, $\mu\alpha\tau\acute{\alpha}$ = 10½ oz. troy).

"[The Arabs] discover gold in underground galleries which have been formed by Nature, and gather it in abundance . . . And since this precious metal abounds in their land, whereas there is a scarcity of copper and iron, they exchange it with merchants for equal parts of the latter wares. DIODORUS SICULUS, *Library of history*, 3, 45.

Of ancient workings, we know more of Southern Spain and Laurion than of others, from their frequent mention by classical historians, as a result of political differences in which they were involved. It has always been a mystery to the author that there are so few remains of ancient equipments in Cornwall, having regard to the great age of the mines; unless indeed the popular idea be correct that they were Phoenician, and did not benefit subsequently from Roman skill in engineering. From Laurion the relics are few but interesting. From Iberia they are many and perfect; interlocking of actual remains with contemporary written and pictorial evidence, and with copies of working drawings in a 8/11th century manuscript, is quite astonishingly complete, as we shall show. The Romans, in general, were gifted civil engineers but in other directions their technical attainment was less than one would expect from a people so skilled and astute in government and administration.



Fig. 1.—Mural from Pompeii illustrating the actual use of the COCHLEA for water raising. This was published in 1927 and is of intense interest as a surviving picture showing the use of the Archimedes' screw not many generations after its invention. (After Halbherr)

1. RICHES OF THE ANDALUSIAN FIELD

Under Roman, Carthaginian, Phoenician and probably pre-Phoenician domination, yield of silver from virgin territories of Andalusia was nothing short of prodigious. In 200 B.C. Lentulus brought back 43,000 *librae* of silver and 2,450 of gold bullion for his triumph in Rome; in 194 B.C. Cato the Elder, apart from specie, exhibited 25,000 *librae* of ingot silver and 1,400 of gold. Many such displays of military spoil are recorded; there can be no doubt that the wealth of Spain was a primary cause for that envy of Carthage by rapacious Rome which led eventually to the Second Punic War. "Turdetania, however, and neighbouring districts are so superior in this respect that the best will in the world cannot express their excellence in words; for gold, silver, copper and iron of similar excellence have not hitherto been discovered in any part of the world"—STRABO, *Geography*, 3, 2, 10.

"The mines being marvellous in their deposits of copper and gold and silver, the workmen in the copper mines recover from the ore they dig out a fourth part of pure copper, and among the unskilled workers in silver there are some who will take out a Euboic talent [c. $\frac{1}{4}$ cwt.] in three days; for all the ore is full of solid silver dust which gleams forth from it."—DIDORUS, 36.

From the quantity of native silver recovered half a century ago at Linares, there is no reason to suspect that this historian's account is anything but strictly accurate. 1 *libra* = 10 $\frac{1}{2}$ oz. troy approx.

PLINY's information clearly came from a different source for he describes a field from which all the concentrated deposits and placer metal had been removed—"Silver is found only in shafts, there being no indications to raise hopes of its occurrence, no shining spangles as in the case of gold. The earth in which it is found is sometimes red, sometimes of ash colour. Moreover it is impossible to melt it except in combustion with lead or with galena, this being the name given to the vein of lead that usually is found running near the veins of silver ore." Surely we are justified in concluding that the native or Roman metallurgists used lead as a collector, and this passage has been quoted repeatedly. Technical deductions from assays of slags and silver-free lead ingots would also support it, and the same conclusion has been reached independently for Greek mining at Laurion.

2. DEWATERING IN ANTIQUITY

Ancient Historical Evidence—

"... but the exploiters of the mines of Spain, in their undertakings, amass great wealth in proportion to their hopes. For their first labours are remunerative thanks to the excellent quality of the earth for this sort of thing, and they continually come across more splendid lodes, rich in both silver and gold, for all the ground in that region is a contorted network of veins, which wind in many ways.

And now and again, as they go down deep they come across flowing subterranean rivers, but they overcome the might of these by diverting the streams which flow in on them by means of water roads leading off at an angle. For urged on by hope of gain, as they are, and



Fig. 2.—Actual Archimedes' screw in oak from Sotiel. (Liverpool Institute of Archaeology).



Fig. 3.—Terra cotta, from Alexandria, (?) showing the COCHLEA in operation. (British Museum)

indeed not disappointed, they push on each separate undertaking to its conclusion, and what is most surprising of all they draw away the waters of the stream they encounter by what is called by the men the Egyptian screw, invented by Archimedes of Syracuse at the time of his visit to Egypt. And by use of such screws they raise the water in successive stages as far as the entrance, drying up in this way the spot where they are digging and making it well suited to the furtherance of their operations. Since this machine is an exceptionally ingenious device, an enormous quantity of water is thrown out, to one's astonishment, by a trifling amount of labour, and all the water from such rivers is brought up easily from the depths and poured out on the surface. And one may well marvel at the inventiveness of the craftsman in connection not only with this invention but with many other greater ones as well, the fame of which has penetrated the whole inhabited world, of these we shall give a detailed and accurate account when we come to the period of Archimedes." DIDORUS *Library of History*, 5, 37. By great misfortune, the books dealing with this have been lost completely; "the craftsman" has been identified with Archimedes.

THE ARCHIMEDES' SCREW OR COCHLEA

VITRUVIUS in his treatise *de Architectura*, devotes a whole volume (X) to what we should term civil and military engineering, including several devices for handling water—the tympanum, or water lifter (Fig. 11) the mill wheel, the screw (Fig. 4) and Ctesibus' water machine, rather like a force pump in principle.

1. "But there is yet a further application of the *cochlea*, which draws a large quantity of water but does not raise it as high as does the wheel. The working of it follows thus—a beam is taken with the same number of fingers in thickness as feet in the length. This is turned to a circle. At the heads the circumference is divided by compass to quadrants and octants, and so in eight parts. Diagonals are lined up so that, the beam being placed horizontal, the lines at each head correspond each to each. The great length is then to be divided up into spaces equal to one eighth of the periphery. Likewise, the beam being laid level, lines are to be drawn from one head to the other guided by a level. Thus equal spaces are marked off on the circumference and on the length.

Where lines are described in longitudinal sense they will make intersections with the cross lines, and in these points are to be marked.

2. "When all these things described have been done, a thin slip of willow or wattle is fixed at the first intersection by a cement of liquid pitch. From there it is drawn obliquely to the intersections of the longitudinal and circumference next following. . . . In the same way slips are fixed obliquely at all separate intersections through the whole distance, and form channels winding through eight divisions of thickness, and a just and natural imitation of a spiral shell (*cochlea*).

3. "Then on this trace, one is placed upon the other, cemented with liquid pitch, and piled up until the thickness is one eighth of the length. Above these, planks are placed round and fixed in order that they may cover the winding part. Then these planks are saturated with pitch and reinforced with iron laminæ in order that they may not be disintegrated by the force of the water. The heads of the beam are plated with iron. But left and right of the screw, fish plates are placed at the heads, having fillets fixed crosswise. In these are included the iron sockets into which the pivots are housed (*inducuntur*) and so the screws are set in motion by human treadmills." Vitruvius' description is clear and exact and such a construction obviously required skill in craftsmanship, a steady hand and great patience in construction. Perhaps it is not too fanciful to see in this *cochlea* the ancestor of the laminated construction so popular for aircraft to-day; Roman willow strips and liquid pitch corresponding to plywood and Bakelite cement. Although Vitruvius wrote in the first century A.D. it is quite clear that some of the mysticism of Pythagoras' number symbolism still persisted; the fixing of the screw was to be at such an angle as corresponded to Pythagorean ratios, e.g., 3, 5 and 4. On a level surface the values could not be otherwise than Pythagorean triads.

The woodcut illustration in Fig. 4 is from Fra Giocondo's edition of VITRUVIUS' *De Architectura*, published in Florence in 1522 in the spate of classics that followed the development of printing from types in most of the flourishing cities of Europe, often by itinerant, or at least semi-mobile, craftsmen. Probably it derives from the 11th century Harley manuscript No. 3859 in the British Museum, the earliest reputable source being the eighth century manuscript No. 2767. At the foot of Manuscript No. 7227 (eleventh century) in the Bibliothèque Nationale is a drawing of the water screw *turris cochlearis*, shown schematically as vertical, with the instruction that it is to be inclined.

Correlation of Evidence of the Age of the Archimedes Pumps from Spain

Our evidence for attributing the screws found in Spanish workings to the Roman period is therefore unusually strong—

1. The rather battered terra cotta from Alexandria(?) showing a slave actually working the apparatus, Fig. 3.
2. The fresh and delightful mural from Pompeii (overwhelmed in 79 A.D.) showing the use of the screw for pumping or irrigation, Fig. 1.
3. The illustration in the early (16th century) edition of Vitruvius, a woodcut doubtless copied slavishly from an earlier manuscript source, Fig. 4.

4. The fact that in some cases the screws themselves correspond closely with the *practical recipe book* description of Vitruvius. See, for instance, Fig. 2 showing the example in oak from Sotiel. In other cases metals (copper and lead) have been employed, an instance of the well-established persistence of archaic patterns after introduction of a new material not suited to them.

When Archimedes, that great research worker and practical craftsman, visited Egypt in 220 B.C., the screw was used for pumping water for irrigation. This continued into the present century; it is still in operation in the Sudan. A painted terra cotta of a slave using this contrivance, probably from Alexandria, and now in the British Museum, is shown in Fig. 3. The workman's hands rested on a pole and his feet operated the inclined barrel like a treadmill, giving an actual head of about six feet and an effective working head about a foot less. About 1927 a wall painting at Pompeii was published by Halbherr showing the *cochlea* in use—Fig. 1.

Cerro Muriano.—RUNDALL has described an Archimedes' screw of oak found at the Cerro Muriano mine of the Cordoba Copper Co.:—5 ft. long and 6-7 ins. in diameter, with barrel of sheet lead. In the same mine, rough preliminary ore dressing was carried out underground on lead tables similar to *planillas* still used in Mexican mines. In the author's view it is likely that neither this nor the metal wood screw from Centenillo described in a subsequent paragraph, is as old as the Roman period.

Sotiel.—The oak screw in Fig. 2, in a remarkable state of preservation, was discovered at the Sotiel Mine and presented to the Liverpool Institute of Archaeology by Dr. G. A. Auden. The central axis is 60.5 cm. (c. 24 ins.) in diameter, the helix projects 6 cm. (2.4 ins.) and is 3 cm. thick. It is built up from slabs 0.3 cm. (c. 1/8 in.) thick, 1-1.7 cm. wide and 3 cm. long, glued together vertically; the pitch at the top of the blades is 17-19 cm.

Centenillo and Castulo Group.—The most perfectly preserved example of a *cochlea* was recovered at the Centenillo mine near Linares. (See *Early Mining in Spain*, METALLURGIA, 33, April, 1946, pp. 293-300, Fig. 13). It consists of a wooden core 8 ins. in diameter, into which a copper helix 1/2 in. thick is fixed. This in turn is attached to the longitudinal laths of the barrel or container, a cylinder 20 in. in diameter and 14 ft.



Fig. 4.—Illustration in a sixteenth-century Venetian edition of Vitruvius' *DE ARCHITECTURA*. It is conjectured that this woodcut was slavishly copied from a much earlier manuscript.



Fig. 5.—Water lifter *in situ* at Rio Tinto, uncovered at the beginning of the present century. (After Palmer)

long. Each end of the barrel was centred in an iron point pivoting in a socket set in the timber. In the Castulo group no less than five of these screw pumps have been recovered, "it is likely that as many as twenty were in use at one time" in the deepest part of the Roman workings.

ROMAN WATER WHEELS

VITRUVIUS describes several kinds of wheel but his account of the type found in mine workings is very cursory—

"But when the water is to be raised to greater head, the same apparatus will be recommended. Thus a wheel is made on an axle of such a size that it can suit the height at which the work is to be done. Round the extreme width of the wheel, square buckets (*modioli quadrati*) caulked with pitch and wax, are to be fixed. Thus when the wheel is turned over by treadmills, full buckets are raised to the summit and returning back to the bottom discharge into a launder (*castellum*) what they brought up." Although he describes a paddle wheel and a mill wheel for grinding, no such types have been reported from workings in Spain. This is reasonable enough, a wheel which requires a current to operate it is obviously quite unsuitable for draining stagnant water from sumps. "Wheels of the same types as that described above are used also in rivers. Round the fronts of the paddles (*pinnae*) are affixed, which, when acted on by impulse of the stream, move forward causing the wheel to rotate; and thus the buckets being filled and carried to the top without the work of tread milling, they are turned over by the impulse of the stream above, which work is useful."

Rio Tinto.—In the northern workings of Rio Tinto a waterwheel with fragments of rope still adhering was found in one of the galleries (*socobones*). Fig. 5 shows the remains of such an apparatus *in situ*, published by PALMER in 1926.

It is a reversed undershot wheel $14\frac{1}{2}$ ft. in diameter with 24 boxes $15\frac{1}{2}$ in. \times 7 in. \times 5 in. deep, fastened by wooden dowels, rotating on an axle of leaded bronze. Probably these wheels were turned out by a sort of rudimentary mass production since they are of identical pinewood and that not indigenous to Andalusia. The hubs are of oak. They were usually placed parallel in pairs, each worked by a slave as a treadmill. By trial on the reconstruction (see Fig. 6) a loaded wheel of 14 ft. 10 in. external diameter was found to balance at 150–160 lb., with an effective lift of 12 ft. PALMER has made a most interesting calculation of the mechanical efficiency of these devices, considered as machines.—

He assumed the total force exerted by the treadmill to be 124 lb. of which 18 lb. is absorbed by axle and paddle friction. For circumferential speed of 30 ft./minute this represents work of 3,720 ft. lb./minute against "accepted data" of 4,200 ft. lb./minute during an 8 hour shift for a convict weighing 140 lb. in the "good old days." On the

reasonable assumption that one fourth of the water raised ran back into the sump, the total output would be $13\frac{1}{2}$ lb. water per minute raised through 12 feet, i.e., 2,270 ft. lb./minute, with overall efficiency 61%.

In 1919–21 a whole nest of these water-wheels was discovered in the Rio Tinto mine. The remains of eight pairs were identified and their arrangement is shown in Fig. 7. To give a constant direction of flow the wheels in each pair revolved in opposite directions and the discharge from each couple was carried to the sump of the pair next above. The engineering involved in this arrangement is very ingenious, but this method of

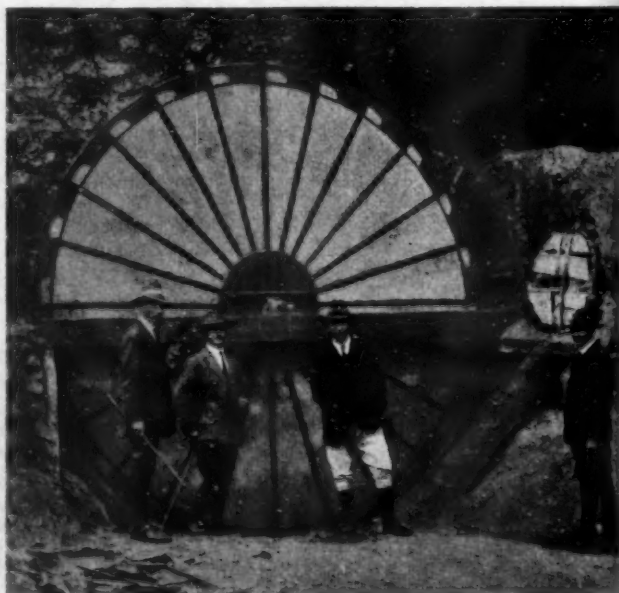


Fig. 6.—Reconstruction of the original wheel in working order. This raised water about 12 ft. (After Palmer)

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dewatering was most inefficient, and costly even with slave labour—eight pairs give a total lift of only 97 feet. VITRUVIUS in describing the apparatus was well aware of its limitations. "However if a supply is needed at places at great height, a double [i.e., endless] iron chain having pendent bronze bowls is wound round the axle of the wheel and let down empty to the bottom. Then rotation of the wheel carries the buckets to the top, the chain winding round the axle; when these are carried over the axle they are made to turn upside down and discharge into the water-laundry what they have raised." Vitruvius seems to have been in first century Rome what we would call a distinguished consulting engineer. It seems strange that a method which he describes so casually should not have been known to mining engineers who were obviously very competent at their own profession. (Figs. 10 and 11 *ibidem*). The author has toyed with the idea that this indicates that

these widen into pumping stations—"In the centre and floor of each room there is placed a square block of granite around which there is a worn circular track such as might have been caused by an animal-driven whim."

In all, about 20 million tons of ore were removed by the ancients, by open-cut and underground working. Assays of vein filling ore ran as follow:—

	%		%		Stops Ore			
					oz.	oz.	oz.	oz.
Cu ..	0.15		present		—	—	—	—
Pb ..	5.5		5.3		—	—	—	—
Ag ..	7½ oz. per		6.8 oz. per		11.9	144.4	57.5	0.60
As ..	oz. 2240 lb.		0.46 oz. 2240 lb.		2½	1.35	0.93	0.78
Fe ..	9.3		8.5		—	—	—	—
SiO ₂	71.5		—		—	—	—	—
Zn ..	1.0		present		—	—	—	—
S ..	5.65		—		—	—	—	—
Sb ..	1.0		—		—	—	—	—

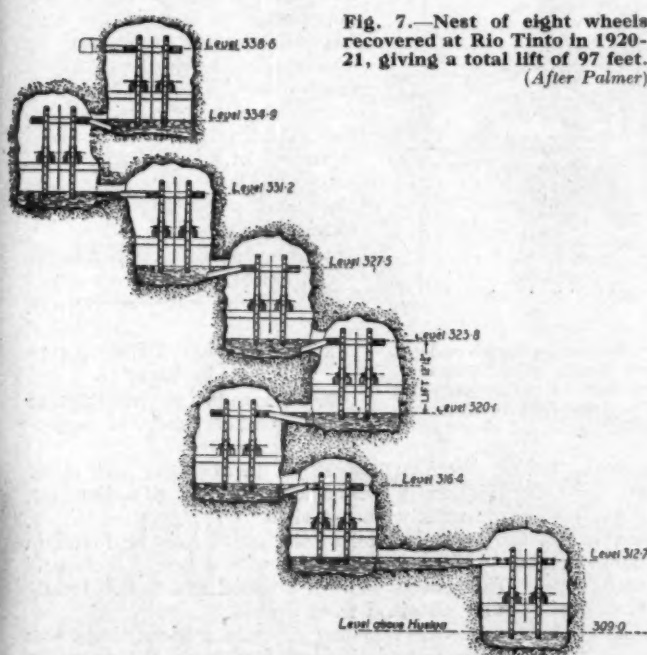


Fig. 7.—Nest of eight wheels recovered at Rio Tinto in 1920-21, giving a total lift of 97 feet. (After Palmer)

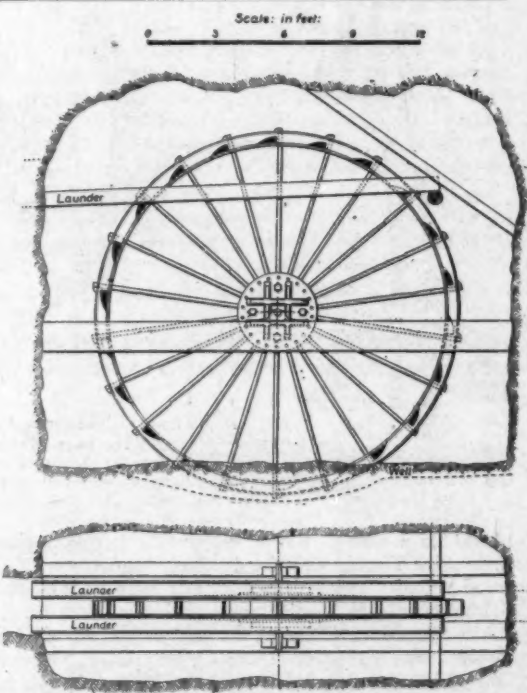


Fig. 8.—Water wheel from San Domingos Mine, Portugal. (After Cross Brown)

the Iberian wheels date from a period before Vitruvius, but in the absence of any data for correlation he would do no more than bring this forward as a suggestion worth the attention of archaeologists. It is astonishing, but nevertheless quite true that the principle described by Vitruvius was transported to the New World by the conquistadores, whence its remains were discovered nearly two millennia afterwards.

San Domingos.—In the San Domingos mine in Portugal nine such wheels in series along a gallery dipping at 40° raised the water to a height at which natural drainage was possible. Altogether over forty such wheels have been recovered from Roman workings.

The skew construction necessary to secure discharge into the outgoing launder, is clearly shown in Fig. 9.

Minas dos Mouros.—In this mine in northern Portugal, HARRISON has described ancient workings, in which narrow water-roads 16 ft. deep have been channelled at the side of broad galleries 16 ft. square. At intervals

One sample taken from an old pillar at a depth of 50 ft., presumably not the richest ore available to the ancients, assayed nearly 3 oz. Au/ton. (See also *ibidem*, Section 3).

This author suggests that the concentration of the ore was much more efficient than smelting. Primitive grinding mills and slags have been found at "dressing floors" near the banks of the torrential mountain streams. Separation was very imperfect, the slags assay 5-7 dwt gold, silver, and enclose shots of lead, especially when coarse-grained.

Vitruvius' Water Wheels in the New World

WOAKES has published a crude water wheel found more than 100 ft. below surface when the Espiritu Santo mine in Darien was re-opened in 1896. This was worked from 1665 to 1727 and possibly in 1777, by the Spaniards. "Five treadwheels were found here, some in a very perfect state of preservation. They were about 12 ft. in diameter and evidently raised the water by stages of

about 30 ft. (?) each. They were all built on the same principle. The small drum about 4 ft. in diameter is mounted on the spokes of the big wheel, and round it the endless chain of buckets is hoisted, the buckets dumped into a dugout launder, inserted under the rim of the small wheel, through which the water was conveyed to the sump of a similar wheel, and again raised another stage. We now know that by this means the Spaniards raised about 80 gallons of water per minute to a height of well over 100 ft." He considered that with two men operating the treadmill the system was rather more efficient than that at Rio Tinto. Fig. 10 shows the

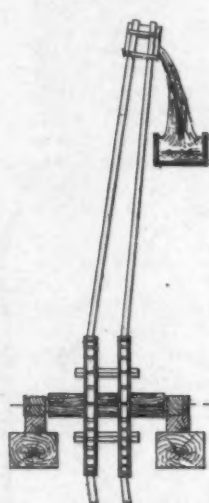


Fig. 9.—Section showing offset discharge into launder.

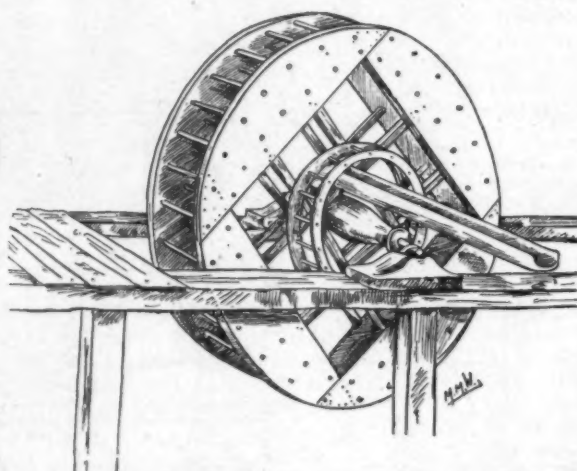


Fig. 10.—Vitruvius' water-lifter (second type with endless chain of buckets) from Espiritu Santo Mine, Panama. This worked under Spanish direction in the seventeenth century. Five of these treadmills, about 12 ft. in diameter, raised the water about 30 ft. each. (After Woakes)

lowest of a series of five, recovered in good repair at Panamá. The outer wheels were connected by hardwood rounds, which formed the treadmill, worked by a pair of men side by side who held stretchers fixed into the walls breast high; an endless chain of leather buckets was constructed like a rope ladder. Round the end of the wooden axle tree iron was used, also in the gudgeon which revolved in a hardwood bush, otherwise the construction was entirely of wood.

According to RESTREPO the mine in Panamá had five ladders of 12–15 steps on entry. There were four sumps and more than 200 men were employed at four levels, the lowest being most important. A royalty of one fifth was paid to the king, and theft by the free negro miners was rampant.

Cyprus and Persia.—A similar contrivance with chain of basket work carrying earthen pots is used in Cyprus for raising irrigation water from the bottom of a well—a horizontal drum rotating on a vertical axis by the power of a mule or donkey moving in a circular track. Moreover the Darien wheel is exactly analogous with primitive wheels still in use in Persia, and closely follows the description given by VITRUVIUS in Volume X.

PRESERVATION OF EQUIPMENT BY CUPRIFEROUS MINE WATER

The very nature of their calling induces engineers to scrap plant as soon as it becomes obsolescent, con-

sequently machinery reaching back so long is rarely found. This is particularly true in excavations, since close contact with soil and moisture plays havoc with equipment, whether of wood or metal. As a result, mining apparatus as old as this, and in good enough repair to be reconstructed, is extraordinarily rare. Since it has been in actual commercial use it is even more interesting than the funeral toys, ceremonial models, stone carvings and wall paintings of Sumer and the Great Kingdoms of Egypt, from which so much has been learned of life in these highly civilised pre-historical ages. That soft pinewood should have escaped disintegra-

tion in damp galleries through twenty centuries, is little short of miraculous; we may reasonably attribute this to bactericidal effect of copper ions in the mine liquor, though one might expect the latter to cause complete disappearance of iron in the end. In 1902 PALMER penetrated an ancient winze at Rio Tinto, 330 ft. below ground level, while driving a crosscut into the slate hanging-wall. At the bottom were found immersed in mine liquor rich in cupric and ferric sulphates, where they had apparently lain for hundreds of years:

1. A bronze shank of a hoe-like tool, with two copper rivets but no [iron?] blade.
2. Several pieces of hemp rope $1\frac{1}{2}$ in. in diameter.
3. Remnants of matting or baskets made of esparto-grass.
4. Five bronze rings, 17 in. in diameter and $1\frac{1}{2}$ in. thick, with holes at opposite ends of a diameter, apparently bucket rims.

In the seventh level of the south lode he found in October, 1900, water-wheels, with money, tools and rope. The wheels were of a wood not found locally and possibly obtained from Galicia.

A bronze miner's helmet of quite modern shape was found in three feet of stiff black leaf mould at the bottom of a 100 ft. shaft in a Roman lead mine near Cordova in Sierra Morena, together with a bronze bell of Celtic type.

VITRUVIUS' DRUM

Vitruvius' description of the *tympanum*, or water wheel with central discharge, is of interest, though it has no relation to the present wheels. Fig. 11, from the same source as the *cochlea*, illustrates the apparatus. "Now this does not raise the water high, but carries away a large quantity very rapidly. [The axle] is worked on the lathe or by the compass, the heads being hooped with iron; having around its own middle a drum of planks fitted together amongst themselves, it is placed on posts plated with iron under the heads of the axle. In the hollow of this drum are placed eight cross planks, tangential to the axle, up to the circumference of the drum which divide the spaces in the drum equally. Round its periphery planks are fixed, openings of half a

foot being left for entrapping the water. Likewise along the axis pigeon-holes are cut in the separate spaces, one to each division. And when, after ship's fashion, it has been pitched, it is turned by treadmill men, and drawing through the openings in the front parts of the drum, [the water] returns through the pigeon holes along the axle into a wooden trough having a channel connected with it."

3. GREEK SILVER-LEAD MINING AT LAURION

Laurion deposits consisted of a mixture of blende, pyrites and argentiferous galena, with normal oxidation at the outcrop; the ancients were interested only in

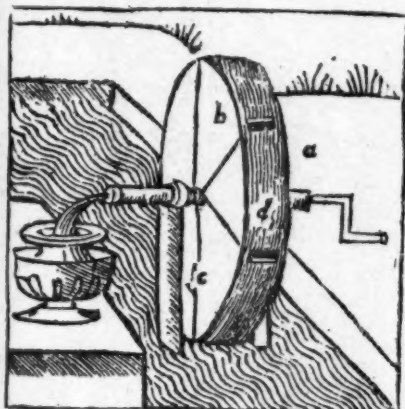


Fig. 11. — Vitruvius' tympanum or drum. Not recorded as having been used in mines.

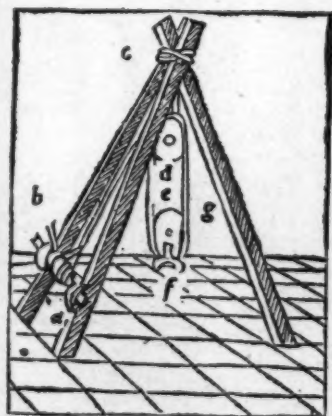


Fig. 12. — Vitruvius' pulley mounted on sheer-legs.

the last mineral. It is clear that initial prospecting soon succeeded in locating the ore at the contact of white limestone and black schist, miners then worked by following the ore body. This has resulted in a veritable labyrinth like those of Andalusia, with occasional caverns where local enlargements of the vein have been worked. Later still, vertical shafts of square or rectangular (but never round) section were sunk, sometimes reaching 400 feet in depth. These were 4 ft. 3 in.—6 ft. 6 in. inside, and divided into sections of 30 ft. each, displaced

through a small angle, giving the effect of a screw when seen from above. It is suggested that this construction was designed to facilitate insertion of wooden battens in mortices in the masonry wall—see also Fig. 7, *ibid.* from Andalusia—leaving the centre free for the passage of baskets carrying ore, or of men. Very tenacious in their determination to reach the mineral lode, the Greeks did not hesitate to abandon shafts and galleries which, to their limited geological knowledge, led into barren ground without hope of striking the profitable vein a second time. A similar follow-your-lode method was in use by miners in Andalusia. A colleague has conjectured that a spade like a hoe or entrenching tool must have been used to dig the vertical shafts. (See *ibidem*, Fig. 5).

It is difficult to assign a date to the beginning of working but the discovery of stone mining tools would suggest pre-1500 B.C. In fact this is no more than the roughest of estimates, liable to amendment as our knowledge grows. Cylindrical stone mortars 1 ft. 9 in. high and about 5 in. thick have been found, also ancient ore mills resembling stone querns from Pompeii. Each consists of a heavy stone annulus rotating on a central hub on a massive central anvil, capable of grinding 4 tons *per diem* to the size of a millet grain, *κερυκός*, whence the name *κερυκός*.

After abandonment they were reopened by Athens about 600 B.C. and a century later their royalty appears in the budget. Controlled ventilation was used, with a fire to induce updraught. Pillar and stall working was usual, and so far as possible the supporting rock pillars were chosen in barren rock or failing that, in lower-grade ore. Where necessary, packing was used to support the

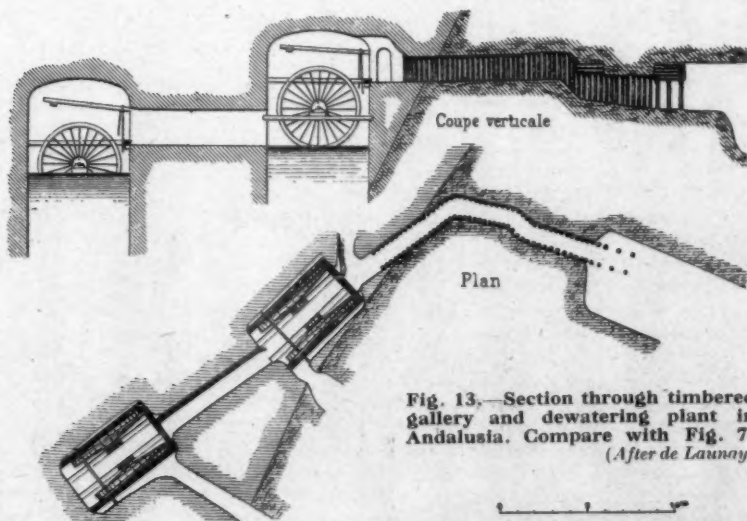


Fig. 13. — Section through timbered gallery and dewatering plant in Andalusia. Compare with Fig. 7. (After de Launay)

roof. It is clear that these pillars must have been broken out in later centuries, by 339 B.C. a law was enacted against this. By the time of STRABO packing was being re-worked and slags re-smelted (1st century B.C.).

The wet processing required an extensive layout of cisterns, flumes, classifiers, etc., and it is conceivable that water supply was a deciding factor in the location of the treatment plant.

Circular smelting furnaces were constructed of mica

schist from Laurion and refractory *trachite* from Milo; according to STRABO they had high chimneys to condense the fume. The condensate consisted of *καδμια*, *σποδος* and *μολιβδος*, possibly zinc oxide and antimony oxide respectively and was highly valued for medicinal purposes. The process was probably not unlike the low furnace treatment of Corinthia in Austria, perhaps even like that in the Scots ore-hearth; from existence of partly altered galena modules in the slags it is clear that the temperature attained was not high. The lead content was 10% or more, by STRABO's time it had dropped, probably to 2-3%. Nevertheless retreatment of old cinder was a profitable enterprise, begun in 1864 and still in operation at the turn of this century.

It is evident that cupellation was carried out very efficiently: contemporary Attic coins assay 980 fine white lead pigs have been discovered running 0.025-0.033 oz./ton. It is fairly clear that this was obtained by reduction of by-product litharge. Its price in the free market was about 11/- (gold) per cwt. in 408 B.C., by 395 B.C. it had dropped to 6/- (gold). Metal price fluctuation, that great bugbear of the producer and user, was obviously as rampant 2,400 years ago as in the inter-war period of this century, and rigging the market just as common. We learn of one speculator buying at 6/- with the object of holding till the price reached 17/-. The lead was used commercially for piping, roofing, and metal manufacture generally. Other by-products were red lead *κινναβρι*, artificial emerald (?), and the medicinal *spodium*.

4. THE COCHLEA IN JAPAN

In 1942 BROMEHEAD published a silk scroll copied in 1840 and depicting a whole gamut of processes in the gold workings of Sado island from ore winning to mintage. From internal evidence the original can be dated post-1637 and post-1639; on external negative evidence it can be attributed to 1647-1697. The scroll shows clearly *tatsudo* or Archimedes' screws introduced by the engineer Soho in 1637. These are slightly conical and obviously of wooden chine construction, the operator sits crouched over the exit end. As in Iberian examples actually recovered, the lift is about seven feet, though some is lost in the discharge of one to the sump of the next above.

How did the Archimedes' pump reach the Orient?

Since the cochlea is not mentioned in *De re metallica* by AGRICOLA (Basle, 1556), or in *Mundus Subterraneus*, by KIRCHER (1664), the invention was possibly unknown in medieval Europe. It is therefore unlikely to have been introduced to Japan by a practical western mining engineer. The author states: "There is the alternative that a Jesuit missionary with a knowledge of the classical writers was responsible. As a personal opinion on what must at present remain an open question I prefer to suggest that the invention reached central or eastern Asia before A.D. 500 . . ." GOWLAND long ago drew attention to the analogies between 19th century Japanese technique in mining and methods abandoned in Europe many centuries before.

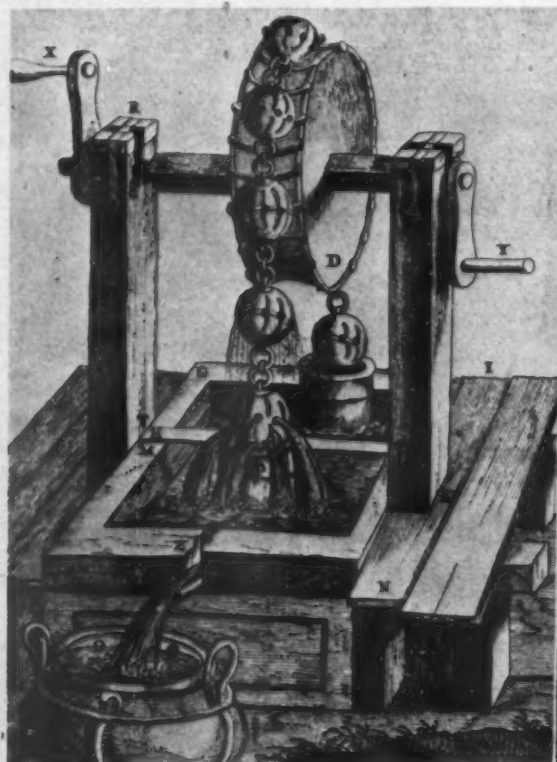


Fig. 14.—Intermittent water-lifter operated manually.

Fig. 15.—Continuous water-lifter operated by treadmill.

Illustrations from Kircher, compare Fig. 15 with Fig. 10, which is approximately contemporaneous.

HYDROGEN

New Uses and Methods of Production

Although hydrogen has, for many years, been recognised as one of the most important of the industrial gases, its wide use in metallurgy is not usually appreciated, yet in this field it has many industrial applications, particularly in the manufacture of comparatively new products, such as metal powders, sponge iron, hard alloys and carbides, as well as in ore reduction and in the treatment of metals. Increasing uses for this gas have led to new methods of production, a brief review of which, together with some of its uses, is given.

HYDROGEN is by far the most important of the industrial gases group, comprising also oxygen, nitrogen, acetylene, the carbon oxides and some others; and its various and manifold uses in the metallurgical industries and indeed in most other fields are constantly extending. These others include the synthesis of organic chemicals, the synthesis of ammonia, nitric acid and nitrates; fat-hardening or hydrogenation of liquid vegetable oils and of whale and other marine oils into hard tallow like fats, of incalculable value to the soap and margarine manufacturer; the manufacture of radio tubes or valves and lamps, and in welding, etc. It is probably, however, in the wide and varied domain of metallurgy that hydrogen finds many of its most important industrial applications—e.g., in the new and rapidly growing manufacture of metal powders, in ore reduction, in the heat-treatment of metals, in the manufacture of sponge iron, and of hard alloys and carbides, and other comparatively new products.

In Europe, notably in Germany, vast quantities of hydrogen have been used for coal, lignite, and tar hydrogenation, and in the U.S.A. for processing hydrocarbons to produce aviation gasoline and lubricants; and these important fields also are likely to be developed on a large scale, to such an extent indeed that other methods of hydrogen manufacture have been introduced in America to augment supplies to an unprecedented degree.

Total production of hydrogen in the U.S.A. to-day has now reached the huge figure of 6 billion (6,000 mill.) cu. ft. per month, of which half is used for ammonia synthesis and about 1 billion cu. ft. for methanol (methyl alcohol) manufacture. One of the principal and comparatively new methods adopted in that country is an improved steam/water gas process. Water gas, consisting mainly of a mixture of carbon monoxide and hydrogen, will react catalytically with steam to produce carbon dioxide and hydrogen; and the latter can be obtained in a relatively pure state by scrubbing the dioxide out of the mixture. This method has been used for many years, especially in ammonia synthesis, but some improvement has been effected in recent years—e.g., in the catalyst employed. This latter now consists usually of iron oxide activated with chromium compounds. Also, the hydrogen losses hitherto sustained in the scrubbing may be avoided or reduced by scrubbing out the carbon dioxide under atmospheric pressure with an amine solution (the Girbotol method).

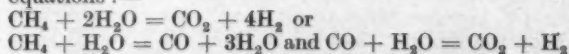
If the hydrogen produced is to be used under high pressure, as in ammonia or methanol synthesis, the dioxide may be removed under pressures of 15–20 atmospheres, and any residual carbon oxides may be removed under still higher pressures, up to 120 atmos-

pheres, if water is used for scrubbing; but these high pressures are not apparently so necessary with the amine solution.

A process of hydrogen manufacture still more extensively used in the U.S.A. and mainly developed during the war is the steam/hydrocarbon method. There are now at least seventeen plants in that country with a total capacity of more than 4 billion cu. ft. per month. Seven of these alone produce 3 billion cu. ft. per month for ammonia synthesis. The principal share in this development is claimed by the Girdler Corporation of Louisville (Girbotol), who further claim a standard of hydrogen purity in their newest type of plant of 99.97%. The hydrocarbon they use is principally propane which is freed from organic sulphur compounds by heating to over 700° F., and passing over a catalyst such as bauxite, to form hydrogen sulphide, which is then removed by caustic scrubbing.

The purified propane is then mixed with steam and passed over a nickel catalyst at about 1,500° F., cooled down to 700° F. by addition of steam at the furnace outlet, and passed through a first stage carbon monoxide converter where the greater part of the monoxide is converted to dioxide and scrubbed out with amine solution. This monoxide conversion plus scrubbing may be repeated once or twice, or even more, according to the degree of hydrogen purity required. The Standard Oil Co., of New Jersey also operates this process on a large scale at their Baton Rouge plant in Louisiana.

Another leading American firm, the Hercules Powder Co., is interested in hydrogen manufacture, and has introduced new methods covered by patents, see e.g. Eng. pat. 2004/1946—open to public inspection. Hydrogen is not only produced by thermal decomposition of natural gas, but also from water, and the possibility of combining these two reactions into one continuous process has long been recognised but not hitherto achieved. It may be represented by the following equations:—



Satisfactory processes are already known for reacting a mixture of gaseous hydrocarbons, such as natural gas, with steam at a high temperature and in the presence of a catalyst of the iron group, preferably nickel, with or without one or more so-called activating substances such as alumina; but with the higher hydrocarbons, and also even with methane and propane, there has been considerable difficulty through carbon deposition. In the Hercules Powder Co's new method the higher petroleum hydrocarbons may be injected at a comparatively low temperature into a current of superheated steam (800°–1,200° F.) in order to heat the hydrocarbons

and the mixture is then immediately subjected to indirect heating, in contact with a suitable catalyst, to the high temperature required for hydrogen production, and without any appreciable carbon deposition.

In the case of liquid hydrocarbons, these may be atomised with steam or other suitable gas, without heating to thermal decomposition point, and the atomised mixture is injected into a superheated steam current. By way of example: three parts by weight of liquid petroleum hydrocarbon, with at least one part by weight of steam, is heated to 300°-700° F. without substantial decomposition; at least four parts by weight of additional steam heated sufficiently (above 1,600° F. or cracking temperature) completely to vaporise the hydrocarbon, in contact with a nickel catalyst, or nickel plus magnesia; and the product—a mixture of hydrogen and carbon oxides substantially free from other carbonaceous material is finally recovered.

It may be added at this point that the steam-methanol process was developed as a portable supply of hydrogen during the war, for which its comparative simplicity and convenience made it readily adaptable; but it is at present rather too costly compared with other processes. Another portable method, apparently designed for wartime exigencies, is that of the British Non-ferrous Metals Research Association, Eng. pat. No. 579246, in which pellets are prepared from magnesium alloy powder (swarf or turnings), powdered iron, or iron oxide, together with a soluble chloride such as ammonium chloride. Hydrogen is obtained by reacting these pellets with water, fresh or salt. The pellets may be prepared from a mixture containing, say, 120 lb. (80% magnesium powder and 20% iron powder) and this quantity will yield about 1,350 cu. ft. hydrogen. A suitable generator of the acetylene type is proposed.

Despite the rapid progress of the newer methods in the U.S.A. the older processes, such as the well-known electrolysis of water and the steam-iron reaction (Lane), are still widely used; and, so far as electrolysis is concerned, the manufacturers are constantly aiming at increased efficiency both in this country and in Canada. Also, their claim to produce the highest purity hydrogen, so essential in many of its uses, especially the metallurgical—up to 99.9%—has not yet been seriously challenged; though the newcomers endeavour to get very near this standard, and if neither time nor expense is spared in supplemental purification operations they may possibly achieve it.

Although the electrolytic processes are well known and have been frequently described, a brief descriptive summary may be included here. They are either: (a) of the uni-polar type, with open or closed tanks, or (b) bi-polar (filter-press) type. Among the best known in the first class are the Knowles (International Electrolytic Plant Co. Ltd., Chester), the Burdett, and the Fauser; whilst the second group is represented by the Levin, the International Oxygen Co's plant, and that of the Consolidated Mining and Smelting Co. Ltd., of Canada. The latter claims to have evolved a highly efficient closed type plant at Trail in British Columbia, in which the c.d. (current density) is 67 amp./sq. ft. and the electrolyte a 28% potassium hydroxide solution. Although one speaks of the electrolysis of water, it is really a fairly strong solution of alkali (soda or potash) that is electrolysed. In the Knowles cell the strength of the alkaline solution is much lower and caustic soda

is used. In this last-named type of cell the purity of the hydrogen obtained is at least 99.9% and of the oxygen 99.5%.

The bi-polar or filter press type was largely used in Europe before the war, among the best known being the Bamag and the Oerlikon (Swiss). The last-named is probably the most progressive in this group, and its makers the Maschinenfab. Oerlikon introduce patented improvements from time to time.

Most electrolytic cells operate under ordinary atmospheric pressure, and although some work has been done on pressure electrolysis in this country, as well as in Germany and India, the method does not appear to have been adopted on a commercial scale to any extent. Normally the hydrogen produced electrolytically needs no further special purification; but in cases where absolutely pure hydrogen (100%) is needed, any small traces of oxygen present—the only likely impurity—may be removed by passing the hydrogen over a suitable catalyst at high temperature.

The Coil Test and V.P.N. Scales

IN the coil test steel bars are passed through a coil with primary and secondary windings, the primary being fed with mains current controlled by special devices while a meter in the secondary circuit gives readings which vary linearly both as regards the diameter of the bar and also of its physical condition.

Although the test is essentially one for machinability and uniformity in the length of individual bars and between different bars of the same batch and consecutive deliveries the records show some degree of linearity between the meter readings and the V.P.N. and Brinell hardness.

Using the formula—V.P.N. equals 640—2.4 times meter reading, the following values ensued for the V.P.N. and calculated values on the coil scale.

(1) On carbon chrome steels treated to get bad structures or received faulty—

	V.P.N.	Coil Scale
(a) Satisfactory as received	200-210	205
(b) Satisfactory as received	197-216	216
(c) Air-cooled from 800° C.	279	278
(d) Air-cooled from 1,000° C.	368	333
(e) Air-cooled from 900° C.	317	324
(f) Hard bar as received	252	246
(g) Just Lamellar	207-224	232
(h) Just elongated Carbides	205-211	235

(2) On miscellaneous steels as received.

	V.P.N.	Coil Scale
(a) Semitool Steel 0.8% C, 0.65% Cr	188	220
(b) Tool Steel 1.4% C, 0.8% Cr, 5% W	221	190
(c) Bright Mild Steel	186	200
(d) Tool Steel 0.7% C, 4.3% Cr, 18% W, 5% Co	289	205
(e) Case-hardening Steel 3% Ni, 0.16% C, 3% Ni	170	173
(f) 0.55% Carbon Steel	237	213
(g) 0.45% Carbon Steel	197	196
(h) 0.25% Carbon Steel	168	175

The field strength used on this test was 30 Gauss. All the bars were 20 mm. diameter.

The figures may be of interest to some of the workers in the field.

THE Ministry of Supply announces that its Malayan Tin Ore Buying Agency (Toba) will cease to purchase tin concentrates in Malaya on December 31st, 1946, and that its Tin Agent in Lower Burma (Mr. A. P. Ruddy) will cease to buy tin concentrates there at the same date. Producers will afterwards be free to sell concentrates to the smelters. The Ministry will, however, continue to buy all the tin metal produced by the smelters in Malaya.

Investigations on Colorimetric Methods of Metallurgical Analysis

*Part II.—A Simple Visual Method for the Colorimetric Estimation of Molybdenum in Alloy Steels

By G. V. L. N. Murty, M.Sc., A.R.I.C., D.Sc.

(Research and Control Laboratories, The Tata Iron and Steel Co. Ltd., Jamshedpur)

A simple visual colorimetric procedure (standard series method) based on Vaughan's photo-electric method for the estimation of molybdenum in alloy steels is described. The results recorded indicate that the new procedure, which is rapid and convenient for routine work, compares very favourably in regard to the accuracy attainable with the gravimetric and photo-electric methods.

AMONG the methods suggested from time to time for the colorimetric estimation of molybdenum, those employing hydrogen peroxide¹ and tannic acid² are not so reliable since the colours involved are susceptible to interference from the presence of iron and other metallic ions. By far, the most satisfactory procedures for this estimation have resulted from the thiocyanate reaction which was first described by Brayn.³ However, the prevailing visual colorimetric methods for the estimation of molybdenum by the thiocyanate reaction prescribe the extraction of the amber colour, developed by the addition of potassium thiocyanate and stannous chloride to an acid solution of the sample, with ether or butyl acetate.⁴ The extracts are compared by matching against standards obtained from known solutions similarly treated. The transient nature of the colour, the persistence of traces of red colour due to iron and the consequent difficulties in effecting colour comparisons have necessitated the use of organic liquids for the extraction. This seems to be responsible for the general disfavour with which these colorimetric methods are held by a large number of routine metallurgical analysts and hence they have failed to replace the time-honoured gravimetric procedures which are always time-consuming and occasionally uncertain.

In the course of our investigations on colorimetry and metallurgical analysis a very simple procedure had been worked out for the visual colorimetric estimation of molybdenum in alloy steels which is being employed by a large number of analysts in our laboratories for the last two years and found to be very convenient and accurate. This procedure is based in its essence on Vaughan's⁵ photo-electric method for the estimation of molybdenum and has resulted mainly from the observation that the colour developed is quite stable for four hours. Small differences in molybdenum

content are readily detectable since the corresponding colour intensities can be easily discriminated with the naked eye. Further, the residual red colour due to iron is more or less of constant intensity and hence does not constitute in any way as an obstacle for the success of the standard series method. The phosphoric acid present in the Spekker acid seems to be exerting a profound stabilising influence on the final colour. Being similar to the recent photo-electric method of Vaughan (loc. cit.) the new visual procedure has the advantage of rapidity and freedom from operations like filtration, extraction, etc. It is, however, necessary that the chromium content should be less than 8%.

Procedure

Reagents required:—

Spekker acid.—Same as in Vaughan's method (loc. cit.)

Mixed reagent.—Prepare the solutions of potassium thiocyanate, sulphuric acid and stannous chloride separately as described in Vaughan's method and stock them. Mix equal volumes of these in a separate bottle and use the mixture for developing the colour. The mixture should not be kept more than two days.

Molybdenum standards:—

Strong standard.—To exactly 15 grams of pure A.R. quality molybdic acid add 75 ml. of strong ammonia, shake for five minutes and warm till a clear solution is obtained. Make it up to 1 litre with distilled water.

Dilute standard.—This is obtained by diluting the strong standard ten times. 1 ml. of this corresponds to 0.10% Mo in the synthetic steel solution described below.

Preparation of standard synthetic steel solutions:—

To 7 grams of ferrous ammonium sulphate (A.R. quality), add the appropriate quantity of dilute standard of molybdic acid followed by 40 ml. of Spekker acid. Boil for five minutes and remove the beaker from the hot plate. Add carefully 5 ml. of dilute nitric acid and boil till fumes of sulphur trioxide appear. Cool and make up to 100 ml.

Preparation of a series of colour standards:—

(These have to be prepared once in every four hours.)

To 5 ml. of the respective synthetic steel solutions add exactly 15 ml. of the mixed reagent and after keeping for 15 minutes transfer to a number of test

* The paper on "Photo-electric Estimation of Nickel in Alloy Steels," Proc. Ind. Acad. Sci. (A), 1945, 21, 73, forms Part I. of this series.

This paper (Part II.) was presented before the Engineering and Metallurgy Section of the Indian Science Congress in January, 1946.

1 Bettel, *Chem. News*, 1908, 79, 40.

2 Spurge, *Chem. Eng. Min. Rev.*, 1919, 11, 258.

3 Braun, *Z. Anal. Chem.*, 1867, 6, 86.

4 King, *Ind. Eng. Chem.*, 1923, 15, 350.

Maag and McCollum, *ibid.*, 1925, 17, 524.

Cunningham and Hammer, *ibid.* (anal. edn.), 1921, 3, 106.

Cox and Pollitt, *J. Soc. Chem. Ind.*, 1944, 63, 375.

Alport, *Colorimetric analysis* (book), Chapman and Hall, 1945, p. 89.

5 Vaughan, The Institute of Chemistry Lecture on "The use of Spekker Photo-electric Absorptometer in Metallurgical Analysis," 1941, pp. 17 and 18.

tubes having the same diameter. The standards usually employed correspond to 0.05, 0.10, 0.15, up to 0.60% molybdenum.

Determination of the percentage of molybdenum:—

Bring 1 gram of steel drillings into solution as described in Vaughan's method (*loc. cit.*). To 5 ml. of this solution add exactly 15 ml. of the mixed reagent and after waiting for 15 minutes transfer to a test tube having the same diameter as those in which the standards are kept. By placing the test tubes very close to the eye and looking through the coloured solutions it can be easily judged whether the unknown is weaker or stronger in colour than the standard. The percentage of Mo in the sample can, therefore, be ascertained by comparing its colour with the respective standards. If the samples contain more than 0.6% Mo it is necessary to take correspondingly smaller quantities. In case the solution obtained is not clear the sample should be dissolved in aqua regia, evaporated to dryness and the residues treated as usual. The results obtained in a few cases are recorded in the following table:—

Per cent. Mo in steel samples (Cr < 8%).

New procedures.	Gravimetric method.	Vaughan's Photo-electric method.
0.08	0.06	0.08
0.15	0.14	0.15
0.17	0.17	0.18
0.22	0.22	0.21
0.34	0.32	0.34
0.38	0.36	0.37
0.43	0.41	0.43
0.52	0.52	0.52
0.56	0.56	0.56

It is readily seen from the above table that the procedure described in this paper compares very favourably in regard to the accuracy attainable with the gravimetric and photo-electric methods. As noted earlier, the colour involved in the method is stable only for four hours and hence the standards have to be developed rather frequently. It would, therefore, be very desirable to increase the stability of the colours for effecting a further simplification in the procedure. Attempts in this direction are in progress.

The author's thanks are due to the management of the Tata Iron and Steel Co. Ltd., for permitting the publication of this paper and to Mr. N. B. Sen, Chief Chemist, T.I. and S. Co. Ltd. for his keen interest in the work.

The Platinum Metals in 1946

DURING the latter part of 1946, platinum use and demand moved rapidly toward normal conditions and new interest developed in other metals of the platinum group, all of which influence Canada directly because of their important production as by-products of the Dominion's nickel output, Charles Engelhard, President of Baker Platinum Inc., stated in reviewing the platinum field in 1946.

The heavy drain on supplies of platinum during the war, when restrictions applied to civilian use and price was fixed by Government order, made the market for platinum vulnerable to speculative trading. Upon removal of price ceilings this speculative trading forced the officially posted prices of platinum to \$90-\$93 which applied for a short period during the latter part of September. At that time weakness developed in the jewellery demand for platinum, which had been maintained at high levels throughout the price rise, and the retail price of platinum subsequently declined to the current level.

Palladium has reached an important position in the industry. Sales of palladium to all United States consuming industries during 1946 are expected to approximate or exceed platinum sales. Platinum and palladium are the two principal metals of the platinum group, which also includes rhodium, ruthenium, iridium and osmium. The United States is the largest consumer of these precious metals.

In jewellery, the white colour of lustrous palladium mirrors the flashing beauty of diamonds which are strongly held. Rings made of palladium are durable as the metal has the strength and hardness to withstand wear. The lightness of palladium is utilised in earrings which are worn with more comfort and also in brooches and other jewellery of large size. Used in the most expensive pieces, palladium is also being employed in moderately priced jewellery and recently has been introduced in the form of palladium-filled costume jewellery and jewellery for men. Chemical and pharmaceutical products including vitamins and hormones, are being synthesised with the aid of palladium catalysts which enable safer processing at lower temperature and pressure. In dentistry, palladium is used to improve the mechanical and fabricating qualities of dental gold alloys for full and partial dentures that give better appearance, comfort and service to the wearer.

Demand for ruthenium increased rapidly during 1946. The hard alloys used for the polished points of writing pens and for similar purposes utilise ruthenium to obtain the high resistance to wear and corrosion required to assure smooth operation over a long period of service. Ruthenium is used also to harden palladium, especially in jewellery alloys commonly made of about 5% ruthenium and 95% palladium.

Rhodium-plated jewellery and optical goods are reappearing in the consumer market, and other rhodium-plated articles including safety razors are in production. Such uses of rhodium were prohibited during the war to conserve rhodium for electroplating mirrors of military searchlights and for industrial and scientific purposes.

Iridium demand in 1946 has largely paralleled the platinum demand for jewellery where a 10% iridium and 90% platinum alloy is in common use. Osmium supplies continue so small that consumption is confined to uses where the total requirements are commensurate with supply. Tiny and highly polished points containing this precious metal are fused to the record-player needles that seldom need to be changed.

The future outlook for platinum metals is promising and the industry looks forward to a period of satisfactory business in markets throughout the world.

Early Mining and Metallurgy

THE Royal Anthropological Institute has appointed a Mining and Metallurgical Committee to investigate problems of early metallurgy, as part of its scheme for group studies of the evolution of man.

Among the first matters to be studied by the Committee is man's early use of copper. When found in its metallic form, the element is known as "native" copper, and metal of this kind undoubtedly formed the earliest sources of copper supply. Samples from various parts of the world are being collected by the Committee and analysed with the object, if possible, of relating their particular composition to that of ancient specimens of copper work.

MICROANALYSIS

CHEMICAL AND PHYSICAL METHODS

APPARATUS, METALLURGICAL APPLICATIONS, TECHNIQUE

THIS is a time of shortages. Our daily life for the past seven years, has, to some extent, inured us to the deprivations which, in many cases, hinder our scientific work. In these columns we have already commented on the difficulties in obtaining apparatus and books. Such difficulties have led many chemists to devise makeshifts whereby their work can be carried on, perhaps with loss of time, but uninterruptedly. However, one shortage which has recently been making itself felt acutely is more difficult to counteract—the shortage of trained personnel. Technicians are necessary in many branches of microchemical work. This is particularly so in organic quantitative analysis, though not peculiar to that type of work. However, it will serve as an example. Many times recently complaints have been made to us by various laboratories that their research staff are continually producing new compounds which are piling up, undeveloped, solely for lack of analyses. It is clear that microchemical training must supply two demands—firstly, the production of research men capable of making the fullest use of microchemical techniques; secondly, technicians capable of satisfying the urgent and insistent demand for routine microchemical analysis. Neither of these is a short-term demand. Time spent in producing either category will by no means be wasted. There has, in recent years, we believe, been more emphasis on the production of the former class, a procedure which is, admittedly, the more difficult. But in a broad survey of demands to be met, the second class must be fully recognised and catered for.

Applications of Metallurgical Micro-chemistry to the Light Alloy Industry

Part II—Special and Investigational Analysis

By Donald F. Phillips, Chief Chemist,
(Research Laboratories, Messrs. High Duty Alloys, Limited)

Routine controls methods which have been handled on the small scale have been dealt with in the previous article. Microchemical analysis for specialised problems requires a different approach. For this purpose a special microchemical laboratory has been equipped, and some description of the problems handled by it is given.

IN addition to production control the analytical division of a light alloy research laboratory is continually called upon to examine materials of a general nature, for example die steels for forging processes, cast irons for gravity die casting moulds, foundry sands, fuels and fluxes. Samples are submitted by the corrosion and anodising departments, and in many cases such samples are available only in minute quantity, e.g., corrosion products, segregates and inclusions in castings or forgings, and quite frequently the analyst is only permitted to remove a few filings or turnings from an article which must not be damaged by drilling or milling.

In order to assess the possibilities of microanalysis with a view to applying the technique to elucidating some of the problems of the light alloy industry, contact was made with two government metallurgical organisations who were known to be making use of microanalysis and facilities were readily granted for an inspection of their microchemical laboratories.

Further advice was sought from a well-known authority who was known to have considerable experience of microchemical methods.

As a result of these enquiries some preliminary trials were made in High Duty Alloys' Laboratories of relatively small scale assays utilising an ordinary good chemical balance to the limits of its sensitivity as recommended by Belcher and Godbert.¹ These enquiries and practical tests not only convinced the writer that microchemical methods would be of great assistance in the field of light alloys, but also indicated the potentialities of certain selected laboratory personnel in regard to their being able to acquire the manipulative ability to carry out such work.

It soon became clear that there were distinct possibilities of linking up the existing systems of spectrography, photometry and polarography with microchemical analysis. It was therefore recently decided to equip a

¹ Belcher and Godbert, "Semi-micro Quantitative Organic Analysis (London), 1945, pp. 8-28.

small microchemical laboratory. As a starting point a room has been temporarily adapted for this purpose, as it is felt that the exact requirements of such a permanent laboratory can only be determined after a certain amount of experience has been gained, and it did not seem advisable to acquire a large amount of general microchemical equipment until precise needs were more fully known. In any case due to manufacturing restrictions many useful pieces of apparatus previously described in the literature by various workers were completely unobtainable, and it was quickly realised that a certain amount of apparatus would have to be home-made. This has not by any means proved a disadvantage as it has enabled the personnel to become acquainted with the art of adaptation, and the devising and preparation of small scale apparatus in the laboratory has provided useful experience.

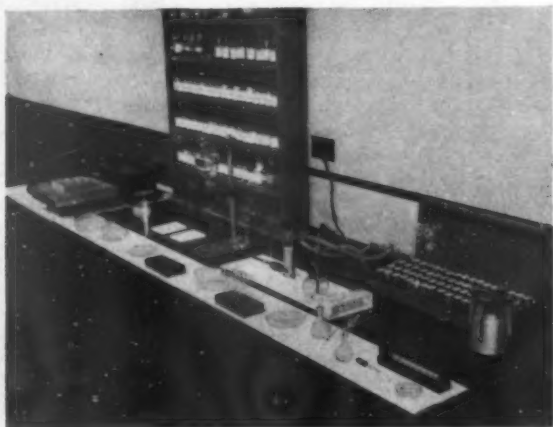


Fig. 1.—Microchemical working bench.

The room selected for the microchemical work is not equipped with gas or water services, but lack of these has not proved a serious handicap in the early stages. Suction for micro-filtrations is provided by a small electrically driven vacuum pump.

The situation of the room, placed as it is within the centre of the laboratory buildings, means that it is well insulated from outside weather conditions, and steep temperature gradients are rarely experienced. Dust contamination and atmospheric pollution are also at a reasonably low level, although when at some future time a permanent microchemical laboratory is designed particular attention must be paid to such details.

An Oertling Type 63PB micro-balance has been installed on a substantial table situated away from the walls, and no special concrete plinth was found necessary as the parquet floor is entirely free from vibrations. As a precaution the table legs rest on laminated sheets of lead. The performance and precision of this balance has been found to be well within the limits claimed by the makers.

An electric hot plate has been adapted from a rectangular platten previously used in a plastic moulding machine. A "Sunvic" temperature control has been incorporated in the circuit.

Coarse or fine control micro burettes as described by

Winteringham² have been constructed and found very satisfactory.

A small electrically driven centrifuge is in use, and is much used for both qualitative and quantitative separations, the necessary centrifuge tubes and capillaries being made as required from glass tubing.

Other devices which are home-made include aluminium heating blocks, as well as cooling blocks consisting of a machined aluminium base in which a circular groove is cut to take a glass Petri dish as the cover.

Although it is considered desirable that the senior analyst, who has shown great enthusiasm and successful application to the development of gravimetric and volumetric micro methods, shall be given a free hand to use any procedure which may appear advisable, it is intended to perfect a scheme of analysis for aluminium alloy samples based on the absorptiometric procedures already described in the previous article. Micro cells and adaptors have now been obtained for using the standard Spekker absorptiometer as a micro instrument.

The polarographic determination of zinc in aluminium alloys mentioned earlier has proved very successful when adapted to micro conditions using a 5 mg. sample, and by the use of smaller polarographic cells this sample size could be still further reduced.

Spectrochemical analysis is of course intimately linked in conception and scale with microchemical analysis, and frequently proves extremely helpful in obtaining a preliminary elucidation of the nature of an alloy or other inorganic material prior to undertaking a complete microanalysis. In the case of metallic substances this can be accomplished either by spark or arc excitation, whilst for non-metallic materials or salts the arc technique is generally necessary.

Physical Methods and Microchemistry

It thus becomes clear that the existing systems of analysis, i.e., spectrography, absorptimetry, and polarography, already in extensive use for control analysis are closely related to true microchemical analysis, and that the experience already gained should prove of immense value in applying all of these techniques to analysis of an investigational nature. Other useful techniques in the analytical field such as chromatography, electrography, internal electrolysis, the mercury cathode, and potentiometric titrations are all readily adaptable to the micro scale, and are by no means being ignored.

Up to the present the view has usually been taken that micro methods are only justifiable where the amount of sample is limited, but it has been statistically proved that quantitative reproduction of chemical reactions on the macro scale can be obtained using amounts of material as small as 10^{-16} g. provided that appropriate techniques of manipulation, observation and measurement are devised.³ This logically leads to speculation as to whether in the future even routine control analysis may not be carried out on a micro scale. The obvious advantages offered through economy of reagents, glassware, and space, are, it is sometimes argued, offset by the increased time and the greater skill required by such methods. The writer's view is that it may be possible to develop such routine micro methods in which there are actually considerable economies of time, and which it will be possible to operate by personnel who, although

² Winteringham, *Analyst*, **70**, No 830, May, 1945, p. 173.

³ Benedetti-Fichler, "Micro Technique of Inorganic Analysis (New York, 1942; pp. 1-4.

chosen with care and discernment for certain temperamental qualities, will not necessarily be required to possess a higher standard of technical education than the average routine analyst employed in industry at the present time. As evidence for this possibility it is only necessary to point out the facility with which many of the operatives (both male and female) who were directed into industrial laboratories during the recent war were able to acquire the art of manipulating spectrographs, microphotometers, absorptimeters, as well as balances, burettes and chemicals.

In the case of metallurgical analysis it may be objected

A Micro H₂S Generator

By A. G. LIDSTONE, C. L. WILSON and D. W. WILSON

NUMEROUS designs have been proposed from time to time in the literature for generators to provide sulphuretted hydrogen for micro and semi-micro inorganic qualitative analysis. Representative types are illustrated in the references quoted.¹

While all of these possess certain advantages, none of them is wholly above criticism, and it therefore seems worth describing a generator which has been used, both for teaching and research purposes, over a considerable period of time, and which has proved very satisfactory.

The principal requirement for such an apparatus is that while in itself small, compact and reasonably sturdy, it must be capable of generating gas at sufficiently high pressure to drive it through a capillary delivery tube in small bubbles, but at such a rate as to prevent blocking of the capillary by sulphide precipitate.

Since the head of pressure is dependent, in most types of generator, on the hydrostatic pressure provided by the hydrochloric acid used in generating the gas, the problem resolved itself into producing a suitable head of pressure while at the same time

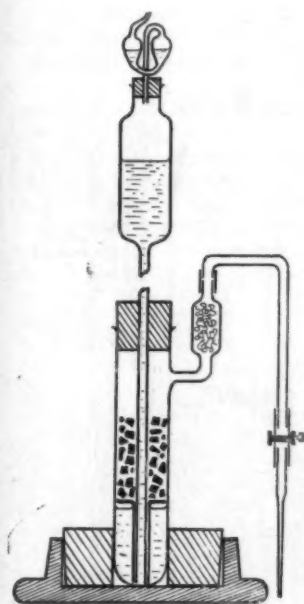


Fig. 1.—Simple form of generator.

keeping down the volume of acid to a reasonably small amount. A development of the ordinary thistle funnel proved adequate for this purpose.

The construction of the simplest form of the apparatus will be reasonably clear from Fig. 1. A modified thistle funnel, which in most of the generators in use was about 20–24 inches high, and with a stem $\frac{1}{4}$ inch in

that the difficulties of obtaining non-segregated samples will prove too serious a difficulty, but this was overcome in the case of metallurgical spectrochemical analysis by taking steps to produce a heavily chilled test-piece in which segregation was reduced to a minimum. In any case microchemical analysis may well prove in the near future to be an invaluable tool for the investigation of segregation in metallurgical products, and to some considerable extent this is already being carried out in the case of light metals at Messrs. High Duty Alloys, Ltd., and has produced information of a most valuable nature.

diameter, fits at its lower end snugly into a short piece of glass tubing which is flared at the top to provide a circular table. The table, in turn, fits closely in a side-arm test-tube, about 1 inch in diameter. Iron sulphide chips are placed on the table—the reservoir can be filled up to the level of the side-arm with these—and the apparatus is then assembled. Rubber tubing on the side-arm is closed by a screw-clip and a delivery tube. Hydrochloric acid (1:2) is then filled in through the top of the apparatus. Escape of surplus gas into the atmosphere is prevented by a trap containing caustic soda solution. The gas is filtered through glass wool in the side-arm bulb, or may, if desired, be washed in a bubbler, before passing into the test solution.

The first model of this apparatus was wired to the back of the bench for stability. Later models used for support a device which, because of its general usefulness merits description here. A large cork, which fits tightly into the hollow of a large inverted glass stopper, is bored to take the side-arm test-tube. When the gen-

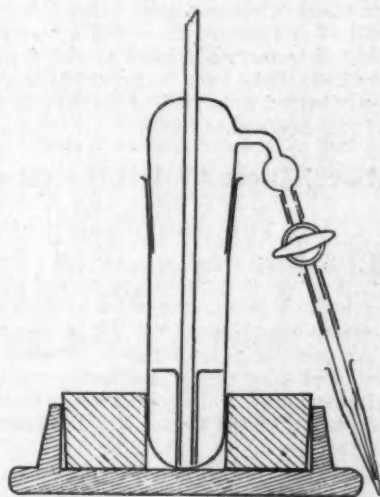


Fig. 2.—Generator with ground-glass joint.

erator is mounted on this as base, it is sufficiently stable to stand without other support. A circular piece of lead sheet inserted first in the stopper will provide an added weight in the base, but is not always necessary. Such bases find use in many directions for the support of filtering tubes and the like, and at the same time offer an admirable method of employing the stoppers from large glass bottles which inevitably accumulate in any laboratory, and which hitherto have not been discarded simply in the hope that "there might be a use for them."

A later model of the generator was made with a ground joint, and its construction, as well as a variation in side-arm, will be clear from Fig. 2.

¹ Cecil L. Wilson, *An Introduction to Microchemical Methods*, p. 93, 1938; S. Gaddie, *J. Chem. Educ.*, 1944, **21**, 101; Anon., *METALLURGIA*, 1944, **30**, 170; Ronald Belcher and Cecil L. Wilson, *Qualitative Inorganic Microanalysis*, p. 8, 1946; Colson, *Analyst*, 1946, **71**, 322.

Yet a third variant provided several delivery tubes, so that it could be used simultaneously by a number of workers.

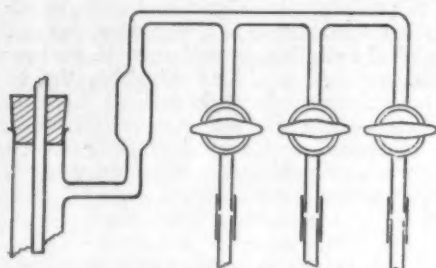


Fig. 3.—Multiple delivery system.

It should therefore be wired appropriately to the shelves at the back of the bench.

In use, generators of this type have proved perfectly adequate for the analysis, in centrifuge cones, of volumes of solution ranging from 0.1 to 1.0 ml. It is advisable to turn on the gas stream before inserting the delivery jet into the test solution. If no bubbler is fitted, it is convenient to use a cone containing the same volume of water for adjustment of the rate of bubbling of the gas.

Spent acid may be drawn off by siphoning, or by using a long glass tube drawn out to a jet and fitted with a rubber bulb. The apparatus will run for a long period on one filling of iron sulphide. When a delivery jet has been used it is removed from the apparatus, rinsed with distilled water, and placed in cleaning acid until again required. Before use it is once again rinsed thoroughly with distilled water. Sir John Cass Technical Institute, E.C. 3., September, 1946.

Miscellaneous Microchemical Devices, X

By J. T. STOCK and M. A. FILL.

A CLAMP FOR OBJECTS OF SMALL DIAMETER

THE clamp shown at (a) and (b), Fig. 1 was designed for use with melting-point and centrifuge capillaries, ignition tubes, etc., which are not easily gripped by a clamp of ordinary form. It is also useful for holding electrodes for potentiometric titration, etc. All parts are of glass or rubber, so that corrosive substances are without effect, whilst operation is instantaneous and is effected with the thumb and forefinger of one hand.

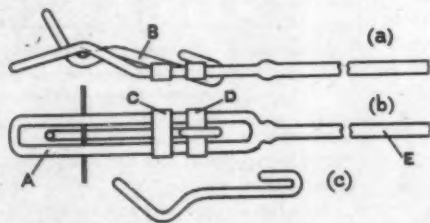


Fig. 1.—Clamp constructed from glass rod.

Fixed jaw A is constructed from a 15 cm. length of 3 mm. dia. glass rod. This is bent hairpin-fashion, the free ends being fused to the end of a suitable length of 4 mm. dia. rod E. A loop of opening 5–6 mm. wide, as shown at (b), is thus formed. The loop is then bent twice at an obtuse angle, as shown at (a). Moving jaw B is

likewise constructed from 3 mm. dia. rod; the shape is shown more clearly at (c). Rubber sleeves C and D act as springs and provide the gripping force. They are cut from 6 mm. bore soft rubber tubing and are about 4 mm. wide. They are slipped on the fixed jaw into the position shown. The hooked end of the moving jaw is then pushed between the sleeves and is engaged upon the rearmost one, as shown. By altering the position of sleeve C, the gripping force may be adjusted.

Provided that the end of the thumbscrew is filed flat, the clamp may be held in an ordinary boss-head. As a precaution, a length of thin-walled rubber tubing may be slipped on E to form a cushion in the boss-head.

Larger clamps, suitable for holding micro-beakers, test tubes, etc., may be constructed from heavier glass rod in a similar manner. Norwood Technical Institute, S.E. 27, October, 1946.

Filtering Devices

TWO variations of set-up for filtration have recently been described by Colson.¹ Their use will be clear from the diagrams.

The set-up in Fig. 1 has been devised for use where barium sulphate precipitates must be transferred to a Neubauer crucible without loss. The upper funnel is ground into its holder to give an air-tight joint. With the apparatus arranged as shown, and the connections air-tight, liquid is held in the upper funnel until suction is applied. It then passes into and through the crucible at a rate which can be easily controlled by the suction.

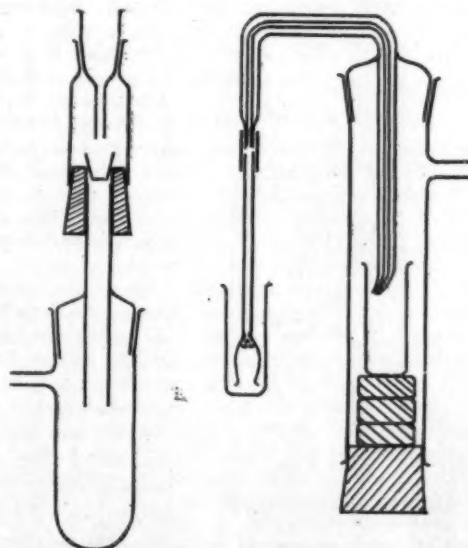


Fig. 1.—Apparatus for controlled filtration.

Fig. 2.—Modified vessel for reception of filtrate.

The second set-up, illustrated in Fig. 2, has as its aim the avoidance of inserting a receiver for filtrate through the top of its container. Instead, it is inserted from the bottom end, and is supported on top of a rubber bung by suitable discs of glass, rubber or wood, at the desired height.

In addition, to avoid the possibility of contamination of the filtrate, the usual rubber bung at the top is replaced by a ground-glass joint.

¹ Colson, *Analyst*, 1946, 71, 322.

METALLURGICAL DIGEST

Partition of Molybdenum in Hypoeutectoid Iron-Carbon-Molybdenum Alloys

By F. E. Bowman

THE effect of molybdenum upon the eutectoid reaction in iron-carbon-molybdenum alloys has been established and investigations have extended to alloys of lower carbon content, wherein the formation of proeutectoid ferrite presents the possibility of another type of partition. The delay of the proeutectoid reaction is essential to complete hardening in hypoeutectoid alloys, and the effect of molybdenum upon this reaction is as important as its effect upon the eutectoid reaction so far as hardenability is concerned. The partition of molybdenum in hypoeutectoid iron-carbon-molybdenum alloys and its relation to hardenability has therefore been investigated.

The selection of the alloy compositions to be studied was determined by the need for sufficient amounts of both the proeutectoid ferrite and the carbide to be present, and it was decided that alloys containing 0.4% carbon were

the most representative of the hypoeutectoid compositions that fulfilled these requirements. The alloys were made from electrolytic iron and had the compositions given in Table I. In addition to the four molybdenum alloys, a molybdenum-free 0.40% carbon alloy was prepared and this alloy was used as a standard for all subsequent work.

All the alloys were homogenized at 1,200° C. for 100 hours to eliminate any molybdenum concentration gradients. Samples were then cut for the determination of the times required for the start and completion of both the proeutectoid reaction and the carbide precipitation at constant subcritical temperature. The molybdenum content of the proeutectic ferrite was determined by the X-ray diffraction method. The ferrite lattice parameters were calculated from X-ray diffraction patterns obtained in a 226.2 mm. diameter back-reflection focussing camera. The molybdenum content of the carbide was determined

by chemical analysis of electrolytically separated carbides.

In carrying out the tests, samples were austenized at 870° for $\frac{1}{2}$ hour, quenched directly into deoxidized lead pots at 705° C. and held for the length of time previously determined for producing as much proeutectoid ferrite as possible without precipitating any carbide. At the end of this time the transformation was abruptly halted by a water quench, and the supersaturated condition of the ferrite which resulted from the quench was relieved by treating for $\frac{1}{2}$ hour at 540° C., followed by slow cooling. The partition in each alloy was investigated in an identical manner after transformation at 650° C. The partition of molybdenum resulting from the tempering of martensite was determined by quenching in water homogenized samples of each alloy from 870° C. and tempering the quenched samples for 150 and 250 hours (*in vacuo*.) The data obtained from the transformations at 705° and 650° C. and from the tempering experiments are also given in Table I.

The results obtained showed that molybdenum segregated in the carbide during the isothermal transformation of austenite at the higher subcritical

From Amer. Soc. for Metals, 1945, Preprint No. 2, pp. 1-16.

TABLE I. COMPOSITION OF ALLOYS AND DISTRIBUTION OF MARTENSITE AFTER TRANSFORMATIONS AND TEMPERING.

No.	Composition		Transformation Times—Seconds			% Molybdenum in						Carbide Structure	Carbide Structure	
			Proeutectoid			Proeutectoid		Ferrite		Carbide			After 150 hours	After 250 hours
	Mo	C	Start	End	Complete	Ferrite	Carbide	After 150 hrs.	After 250 hrs.	After 150 hrs.	After 250 hrs.			
DISTRIBUTION OF MOLYBDENUM AFTER ISOTHERMAL TRANSFORMATION AT 705° C.														
2	0.24	0.41	15	60	480	0.24	0.75	—	—	—	—	Orthorhombic Orthorhombic + Face-centered cubic		
3	0.48	0.38	35	360	800	0.47	1.78	—	—	—	—			
4	0.68	0.41	50	630	3600	0.67	5.09	—	—	—	—	Face-centered cubic		
5	0.93	0.41	80	1000	7200	0.93	7.07	—	—	—	—	Face-centered cubic		
DISTRIBUTION OF MOLYBDENUM AFTER ISOTHERMAL TRANSFORMATION AT 650° C.														
2	0.24	0.41	5	35	400	0.23	1.19	—	—	—	—	Orthorhombic Orthorhombic + Face-centered cubic		
3	0.48	0.38	15	400	850	0.48	1.98	—	—	—	—			
4	0.68	0.41	300	820	2400	0.67	4.10	—	—	—	—	Face-centered cubic		
5	0.93	0.41	420	2400	6000	0.93	6.25	—	—	—	—	Face-centered cubic		
ALLOYS QUENCHED FROM 870° C. AND TEMPERED AT 705° C.														
2	0.24	0.41	—	—	—	—	—	0.17	0.15	1.15	1.50	—	Orthorhombic Orthorhombic 90% Orthorhombic	Orthorhombic Orthorhombic 90% Orthorhombic
3	0.48	0.38	—	—	—	—	—	0.34	0.31	2.31	2.81	—		
4	0.68	0.41	—	—	—	—	—	0.48	0.44	3.43	4.01	—	10% Face-centered cubic	10% Face-centered cubic
5	0.93	0.41	—	—	—	—	—	0.62	0.58	4.79	5.33	—	80% Orthorhombic	80% Orthorhombic
													20% Face-centered cubic	20% Face-centered cubic

temperatures. In alloys containing more than 0.50% molybdenum and isothermally transformed at 650° and 705° C., the cementite was replaced by an iron-molybdenum carbide ($\text{FeMo}_{23}\text{C}_6$) having a larger and more complex unit cell. Molybdenum also retarded the rate of formation of proeutectoid ferrite, although the molybdenum content of the ferrite remained unchanged from that of the austenite, and molybdenum moved from ferrite to the carbide during the tempering of martensite, and the rate of graphitisation during such tempering was retarded by molybdenum. The molybdenum concentration in the orthorhombic carbide (Fe_3C) could be increased by tempering martensite to a value greater than that which resulted in the presence of the face-centred cubic carbide ($\text{FeMo}_{23}\text{C}_6$) after

the isothermal transformation of austenite.

The segregation of molybdenum in the carbides formed by the isothermal transformation of austenite at 705° and 650° C., demonstrated the diffusion of molybdenum during the eutectoid transformation process. The delay effected by molybdenum in the eutectoid reaction was accounted for by the slow rate of diffusion of molybdenum as compared with that of carbon, which determines the rate of transformation in unalloyed austenite. In addition, under conditions favouring a relatively high molybdenum concentration in the carbide, the rate of carbide nucleation and growth was further decreased by the formation of a large and complex face-centred cubic carbide of iron and molybdenum.

Effect of the Iron Content of Cupro-Nickel on its Corrosion Resistance in Sea Water

By A. W. Tracy and L. R. Hungerford

DATA are given of a laboratory investigation concerning the effect of iron additions to cupro-nickel alloys on the corrosion resistance of the alloys exposed to "sea water" in motion. Tests were made both in a vertical-shaft impingement tester and in a horizontal-shaft impingement tester. Test specimens in the form of strips, 1 in. wide by 0.040 to 0.050 in. thick and in lengths varying from 2 to 6 in., depending on the velocity required, were fastened to fibre discs by

means of Everdur bolts suitably protected. Sixteen tube specimens of copper-nickel alloys, 61 in. long by $\frac{5}{8}$ in. outside diameter by 0.049 wide, spaced around an 8 in. circle in Everdur tube plates, were also tested in an experimental condenser. The "sea water" used in the test was a 3% solution of sea salt, and the degree of corrosion was determined by measuring the thickness of specimens before and after test, using sharp-pointed micrometers.

Duplicate specimens of copper, plain Admiralty brass, arsenical Admiralty

brass, aluminium brass, 30% cupro-nickel with low iron, 30% cupro-nickel with 0.45% iron, and a special 10% nickel silver were first tested on the vertical-shaft impingement tester for 26 weeks at room temperature, at a peripheral velocity of 8 ft. per sec. The losses in thickness for these tests are given in Table I. Single specimens of 30% cupro-nickel containing up to 1.5% iron and a short series with 0.5% iron and varying manganese content, were tested on the vertical-shaft tester for 12 weeks, at room temperature, at 20 ft. per sec. A third series of vertical-shaft tests was carried out in which 10 and 20% cupro-nickel alloys with iron contents ranging up to 1.5 were compared with two 30% cupro-nickels, one substantially free from iron and the other with 0.5% iron, for 8 weeks, at room temperature, at 26 ft. per sec. Two series of tests were also carried out with 10 and 30% cupro-nickel alloys, one in the vertical tester for 8 weeks at room temperature, at 26 ft. per sec., and the other in the horizontal tester for 18 weeks at 40° C. at 48 ft. per sec. In view of the excellent resistance of 10% cupro-nickel alloys, containing substantial amounts of iron, tests were made to determine the effect of iron in amounts up to 1.5% on the corrosion resistance of 2% and 5% cupro-nickel alloys when exposed to sea-salt solution at high velocity. A direct comparison test was also made with the vertical-shaft impingement tester on 2, 5, 10, 20 and 30% cupro-nickel alloys.

The results obtained from the tests showed quite conclusively the effect of iron content in improving the resistance of cupro-nickel alloys to impingement corrosion by sea water. The greatest improvement was imparted to cupro-nickel alloys containing between 10 and 20% nickel. The alloy with 5% nickel was improved to a fair degree by the addition of iron and the alloy with 2% nickel showed some improvement when iron was added. Approximately 1% iron had no effect in improving the resistance of copper to sea-water impingement corrosion. The tests also indicated that, as the nickel content of cupro-nickel alloys decreased from 30 to 5%, more iron is required for optimum corrosion resistance, for example, 0.5% iron for 30% cupro-nickel, 0.6% iron for 20% cupro-nickel, 0.75% iron for 10% cupro-nickel and 1% iron for 5% cupro-nickel. There was no indication that a critical relationship between the iron and manganese contents of cupro-nickel alloys affected corrosion resistance.

From *American Soc. for Testing Materials*, 1945, Preprint A3, pp. 1-23.

TABLE I. ALLOYS TESTED IN VERTICAL SHAFT IMPINGEMENT TESTER FOR 26 WEEKS AT ROOM TEMPERATURE, RUN AT 8 FT. PER SEC.

No.	Alloy	Composition							Maximum Penetration Ins.	Remarks
		Cu	N	Fe	Mn	Zn	C	Other Elements		
1	Copper . . .	99.960	—	—	—	—	—	P 0.033	0.017 0.022	No visible film
2	Admiralty Plain . . .	70.58	—	0.01	—	28.33	—	As 0.004 Pr 0.02 Sn 1.06	0 0	Green film
3	Admiralty Arsenical . .	70.71	—	0.03	—	28.08	—	As 0.03 Pb 0.01 Sn 1.15	0 0	Green film
4	Aluminium Brass . .	76.42	—	0.07	—	31.46	—	As 0.056 Al 1.98 Pb 0.01	0.010 0.007	Thin green film except in pitted areas
5	30% Cupro-Nickel	69.02	30.39	0.05	0.29	0.50	0.042	Pb 0.01	0.025	Thin green film
6	20% Cupro-Nickel and 0.5% Iron	68.09	29.92	1.20	1.20	0.30	0.40	—	0.001 0	Dark green film
7	Special 10% Nickel Silver . . .	56.61	10.50	0.06	0.06	32.73	—	Pb 0.012	0.008 0.010	Grey-green film

Super Alloys for High-temperature Service

By H. A. Knight

SOME 40 high-temperature alloys have been used in the United States since 1942, of which at least 35 were designed to withstand temperatures of 650°-870° C. (1200°-1600° F.), and the other 5 in parts somewhat removed from the most severely heated zones. From a tonnage standpoint the bulk of the high-temperature metals or super alloys have entered, firstly, into the production of turbosuperchargers, and, secondly, into the production of aeroplane jet engines. Side by side in the development of turbosuperchargers and jet engines has been that of steam turbines and more particularly gas turbines for stationary power or heavy mobile power equipment. In the jet engine or stationary or marine gas turbine, expansion can be taken care of in many parts by special expansion joints, but the main problem from a metallurgical standpoint

has been to find alloys for the blades and wheels that would withstand high temperature and pressure without expanding and distorting.

There are two important and somewhat diverse concepts on the functions of high-temperature metals. Where war equipment is involved, high cost and short life are allowable, so that turbosuperchargers and jet engines need not last too long. The gas turbine used as a power plant or mobile unit, however, must be designed to last for a number of years. Tests and service failures have shown that the highest quality of metal is demanded from the blades which are the moving parts of a turbine. Almost as severe punishment must be undergone by the wheels or discs. Nearly equal quality is demanded of stationary parts such as vanes, diaphragms or nozzles. What may be termed super-super alloys are those used in a turbine for blades and discs.

In choosing high-temperature alloys certain important metallurgical considerations are necessary. Chromium provides corrosion and oxidation resistance and molybdenum is considered the most effective element for high-temperature strength. To balance the chromium and molybdenum and assure a purely austenitic steel free from delta iron, which would lower the high-temperature strength and cause low ductility on ageing at operating temperatures, an addition of 25% nickel is required. Low carbon is required for good forging and welding. Tungsten, vanadium, titanium and columbium are inclined to introduce melting difficulties. Nitrogen stabilises austenite and nitrides have greater resistance towards spheroidisation at elevated temperatures and aid creep strength. Increased manganese improves forgeability and silicon is an aid to scale resistance.

An alloy containing 28% chromium is the most corrosion- and heat-resisting one available, but develops undesirable mechanical properties because of changes in constitution during heating. A brittleness develops at 480° C., which is relieved by heating

From *Materials and Methods*, 1946, Vol. 23, No. 6, pp. 1557-1563.

TABLE I. HIGH-TEMPERATURE ALLOYS.

Alloy	C	Mn	Si	Cr	Ni	Co	Mo	Cb	W	Fe	Others	Maker	Parts used in
16-25-6	0.08	1.50	0.80	16.50	25.00	—	6.25	—	—	Bal.	—	Timken Roller Bearing	Wheels.
17-W	0.30	0.60	1.00	13.00	30.00	—	0.60	—	2.25	Bal.	—	Universal Cylcops Crucible Steel Co. Midvale Steel Co.	Turbo-supercharger Blades and Wheels.
Gamma Columbian ..	0.40	1.00	1.00	15.00	25.00	—	2.00 4.00	4.00	—	Bal.	—	Allegheny Ludlum Universal Cylcops	Turbo-supercharger Wheels.
E.M.E.	0.10	0.75	0.50	19.00	12.00	—	—	1.25	3.25	Bal.	N 0.15	Midvale Steel Co.	Large Disc Forgings for Gas Turbines (in development stage).
8-816	0.40	0.50 0.75	0.50 0.65	20.00	20.00	44.00	4.00	4.00	4.00	Bal.	—	Allegheny Ludlum	Wheels.
Discalloy	0.04	0.50	0.50	13.00	24.50	—	3.00	—	—	Bal.	Ti 2.25 Al 0.60	Westinghouse	Wheels.
K-42 B	0.05	0.70	0.40	18.00	42.00	22.00	—	—	—	Bal.	Ti 2.00 Al 0.60	Westinghouse	Turbo-supercharger Blades.
Hastelloy B	0.05	0.60	0.25	—	65.00	29.00	—	—	—	4.50	—	Haynes Stellite	Blades.
Vitalium	0.30	0.30	0.25	27.00	2.00	64.00	5.00	—	—	2.00	—	Anstetal Laboratories	Turbo-supercharger and Jet Blades, Nozzle Vanes, Liners, Tail Cones.
Inconel	0.15	1.00	0.50	12.00 15.00	75.00	—	—	—	—	9.00	Cu 0.50	International Nickel	Liners of Combustion Chambers.
Vanad. Corp. N.D.R.C. Research	—	—	—	25.00 35.00	20.00 30.00	20.00 30.00	—	—	15.00 25.00	—	B 0.25/1.00	Vanadium Corp.	—
Climax Moly. N.D.R.C. Research	—	—	—	60.00	—	—	15.00 25.00	—	—	15.00 25.00	—	Climax Molybdenum	—
X-40	0.40 0.50	—	—	25.00 28.00	10.00 12.00	54.00 55.00	—	—	7.00 7.50	0.60	—	General Electric	Gas Turbine and Jet Engine Blades.

to 815° C. and cooling rapidly. During service at 656° to 925° C. nothing unusual occurs if cooling below 650° C. is rapid. At 925° C. and higher, large grain growth results in brittleness. Only 16% chromium is required to resist oxidation from 700°-815° C. but 27% is required between 815° and 1,175° C. The higher the chromium content, the greater the concentration of chromium oxide and the less the scaling, but as the carbon content increases to 1%, resistance to scaling decreases.

Most austenitic steel carburises at over 700° C., but the addition of silicon to the 25-20 chromium-nickel steel brings about a marked reduction in tendency to become carburised and with over 1.7% silicon there is no carburisation. The 28% chromium steel which is ferritic at all temperatures resists carburisation better than the austenitic steel, and the non-hardening 12% chromium steel, with aluminium and low carbon, which remains ferritic up to 925° C., also resists carburisation, although it can be carburised at 1,040-1,100° C. The 18-8 chromium-nickel steel, containing molybdenum, is the strongest of the regular grades of stainless steel at high temperatures.

Some of the high-temperature alloys used in the United States during recent years are given in Table I. In the manufacture of turbosupercharger wheels or discs, 17-W was first used in 1937 and in 1941 gamma columbium was introduced, though approved usage was very small and there was no production in quantity. 17-W has good rupture characteristics and some ductility. Gamma columbium has good rupture characteristics but difficulties were encountered when welding the blades to the wheel and in drilling and reaming. Timken 16-25-6 was also adopted for discs during 1941 and used during most of the war, and E.M.E. was developed later in the war and used for large disc forgings for gas turbines. K-42B and Discalloy were confined to development units and never used in production.

Vitallium and Hastelloy B have been used for jet-engine blades. Since the latter alloy contains no chromium, its resistance to oxidation is poor and it has to be spheroidised at 1,040° C. for 24 hours before going into service. A more recent and most successful blade material is S-816 which showed good rupture strength where used in one design. For jet engine liners for the combustion chamber most successful alloys used were Inconel, an alloy containing 25% chromium, 20% nickel and 2% silicon; and Vitallium. Near

the close of the war, two high temperature alloys were developed, Climax Moly N.D.R.C. Research and Vanadium Corp. N.D.R.C. Research, both of which showed unusual promise. The former contains 60% or more chromium

and has a high-rupture strength but is inclined to brittleness. The latter contains almost equal amounts of chromium, nickel, cobalt and tungsten, to which is added a trace of boron.

Engineering in an Atomic Era

By A. C. Klein

NO one, who has been present at the detonation of an atomic bomb and who has witnessed the awe-inspiring devastation that has followed, can have any doubt that we have just turned over a leaf in the book which records the progress of civilisation. Except for a few smudges, it is a clean page on which we may write a history of the attainment of peace and plenty, or of the greatest holocaust that mankind has ever known.

Power is the index of civilisation. Adequate power for fertilisers, for irrigation, and for farm machinery together spell a prosperous agriculture and freedom from hunger and famine. Adequate power for mines, for factories and for transportation spell freedom from industrial slavery and low living standards and, incidentally, adequate power for household uses will go far towards ensuring domestic peace.

In considering the future of atomic energy its military aspects cannot be overlooked. All are vitally interested in seeing that the international situation is cleared up as early as possible. But it is in connection with its peacetime application that mankind is primarily interested and many questions are asked concerning this aspect. The most common of these questions are: How soon will it come? How extensive will its application be, and what will be its effect on power costs?

The task of producing atomic energy and of converting it into electricity or steam is a task equal in magnitude to that of constructing one of the major atomic-bomb-material plants, and the time that will be required to do so will be of the order of from three to five years. Moreover, after the project is solved from an engineering standpoint, it will not necessarily be economically sound. Only continued operation of atomic-energy plants for a period of many years, and the improvement in design and operating techniques that will come through such operation, can result in what we commonly term "cheap" atomic-power energy. It will be at least ten years before atomic

energy will have any effect upon electricity costs.

Atomic energy will supplant coal and other fuels to a limited extent only, because there is room enough in the growth of demand for power to absorb all of the uranium that will be produced and at the same time to keep coal mines, oil wells, and hydroelectric plants operating at normal capacity. The effect of atomic energy will be minor compared to the fluctuations in fuel demands that accompany changes in business cycles.

The industries likely to generate atomic energy to best advantage are the very largest consumers of power. The atomic-energy unit is inherently a large-scale device. Operation is accompanied by continuous emission of radioactivity. Operators and property must be protected by walls of concrete, usually about six feet thick. Chemical-separation facilities must be provided so that the active material may be reprocessed periodically to remove the fission products which after a time slow down or stop the operation. All of these are factors which will preclude the development of atomic energy on any small scale.

The first commercial plants to be built would be of the order of hundreds of thousands, perhaps even up to a million kilowatts in size, that would be connected to an interconnected network of several of our largest public utility systems, and industry and the public in general would be affected very little by their operation, except that an ultimately lower cost of producing electricity will make it possible to lower electric power costs. At a later time, the electrochemical industries will undoubtedly have their own atomic-power plants but, in the main, industries will continue for a long time to receive their atomic power over the lines of their electric-power utilities.

Another important peacetime use of atomic energy is in the production of radioactive materials for medical and biological purposes. This work is now under weigh on a semi-commercial basis.

From Mech. Eng., 1946, 69, 1029-1031.

An important outgrowth of the atomic-bomb is in the field opened up for the separation of isotopes. Isotopes are forms of an element which differ principally in their weight. They do, however, differ very slightly in other respects, and it is quite probable that

their separation and study may lead to important developments in chemistry and metallurgy. Isotope study is a practically untouched field and much work lies ahead for scientists and engineers in it.

The Effect of Overstress in Fatigue on the Endurance Life of Steel

By J. B. Kommers

IN three previous papers, the author has discussed the effect of overstressing in fatigue. In those experiments a stress above the endurance limit was applied for a number of cycles less than that necessary to cause failure, and the damage caused by the overstress was measured by the percentage that the new endurance limit had been decreased as compared with the original endurance limit before overstress had been applied. Since endurance life is an important consideration in many applications of

The two materials investigated were an S.A.E. 1020 steel containing 0.20% carbon, 0.37% manganese, having a yield point of 17.4 tons per sq. in., an ultimate stress of 26.0 tons per sq. in., an elongation of 44.5% and an endurance limit of 12.5 tons per sq. in.; and a high-strength steel B containing 0.30% carbon, 1.50% manganese, 0.46% copper, having a yield point of 25.0 tons per sq. in., an ultimate stress of 42.2 tons per sq. in., an elongation of 31.5% and an endurance limit of 20 tons per sq. in. The 1020 steel was received in the form of $\frac{1}{2}$ in. round bars, and steel B as $\frac{3}{4}$ in. round bars, and both steels were tested as received. The fatigue tests were carried out on R. R. Moore fatigue machines, which

runs of 48,000, 72,000 and 96,000 cycles. In Table I are given the new endurance limits after the various cycle ratios at 29% overstress. The combinations of overstress for steel B were 30% initial and 10% final, 30% initial and 20% final, and 20% initial and 10% final. Results were obtained for S.A.E. 1020 steel when the initial overstress was 5% above the original endurance limit, and the final overstress was 10, 15, 20 and 25%, Fig. 1. Three damage curves were also obtained for Steel B, Fig. 2.

TABLE I. DAMAGE OF ENDURANCE LIMIT

Cycle Ratio %	New Endurance Limit. Tons per sq. in.	Damage to Endurance Limit %
20	11.6	4.7
40	11.5	7.2
60	11.2	10.0
80	10.9	12.5

The results obtained showed that a high initial overstress for a given cycle ratio followed by a lower overstress might, in some cases, show equal damage to endurance life at the high and low stresses. In other cases the higher stress might cause a greater percentage of damage at the lower stress. A low initial overstress for a given cycle ratio followed by a higher overstress generally showed that the percentage of damage to endurance life at the lower stress caused a small percentage of damage to endurance life at the higher stress. Low initial overstress, for small initial stresses and the lower cycle ratios might produce actual increase of the normal endurance life at a subsequent higher stress. For both the first two combinations of overstress mentioned, steels of higher tensile strength and lower ductility were more sensitive to damage than the weaker steels with greater ductility. If the facts found are generally true for all steels, it might be assumed that a low initial stress will not cause more than equal damage to endurance life at a subsequent lower stress.

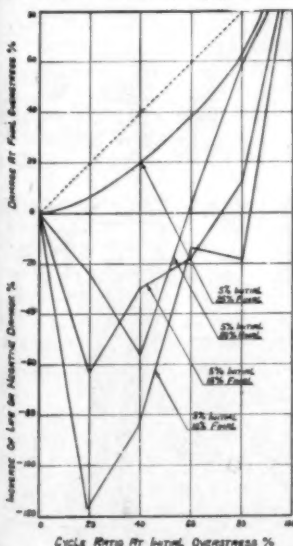


Fig. 1.—Damage curves for S.A.E. 1020 steel.

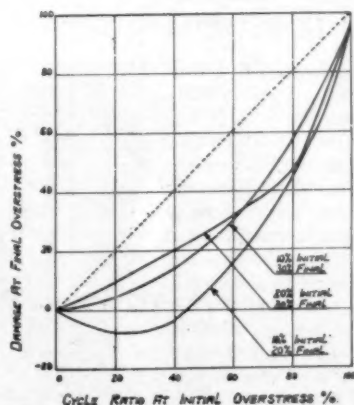


Fig. 2.—Damage curves for steel B.

materials subjected to fatigue, it appeared desirable to determine the effect that overstressing produces on endurance life rather than on endurance limit. In the tests carried out the damage caused by an initial overstress was measured, therefore, by the percentage that the endurance life at a final higher or lower overstress was decreased as compared with the normal life at the final overstress.

subjected the rotating-beam specimens to completely reversed bending stresses at a rate of 1,750 cycles per minute.

In the first series of tests on the 1020 steel, the initial overstress was 16 tons per sq. in., which was 29% above the original endurance limit, for a cycle ratio of 20%, which was 20% of 120,000 cycles, the average normal life at this stress. Results were also obtained for an initial overstress of 40, 60 and 80% corresponding to initial

Aluminium-Bronze Permanent Mould Castings

By B. Johnson

ALUMINIUM-BRONZE castings made in permanent moulds are produced to unusually close tolerances and have smoother surfaces, higher hardness, and superior grain structure, than comparable sand castings. Those

From *Proc. Amer. Soc. Testing Materials*, 1945, Vol. 45, pp. 532-541.

From *Iron Age*, 1946, 157, No. 14, p. 80.

produced at the works of McGill Manufacturing Co. are cast exclusively of an aluminium bronze alloy of the following analysis: Cu 85 to 88%, Al 10 to 11%, Fe 2 to 3%. Physical properties of these castings show a tensile strength of 75,000 to 85,000 lbs./sq. in., a yield strength of 36,000 lbs./sq. in., an elongation of 17 to 26%, and hardness of Rb 72 to 85 as cast. Higher hardness values can be achieved by heat treatment.

This aluminium-bronze alloy is used exclusively because it has been found to be the only copper-base alloy having the high fluidity considered necessary for this type of casting. Other bronzes and brasses do not flow freely enough for gravity pouring into metal moulds which, although kept hot, still exert a decided chilling action on an alloy that has a melting point of 1,870° F.

Gravity-filled permanent moulds such as are used in this plant are always made in two or more parts and must be held together by mechanical means. Comparatively simple and inexpensive hand-operated machines are employed for this purpose. The main

parting of most permanent moulds is in a horizontal plane but the lower block of the mould or die usually is split to clear a runner, while the upper die is pulled out or off the casting and any lower slide or core used is broken loose.

When long cores are required, they usually project through the upper half of the die and are pulled by a lever-operated rack and pinion. Because of high shrinkage, a considerable force is required for core pulling. Inserts, when used, usually project into the upper die block. Some very simple dies have a single vertical parting through which an insert may project.

Dies are preheated each morning to expel moisture and to bring to a favourable casting temperature. This temperature is maintained, as long as casting continues, by heat from the cast metal. Dies are filled through an angular sprue hole in the die parting, with the metal flowing from the sprue hole through a runner and gate and thence upward to fill the die cavity. Runners and gates have to be arranged so that they clear the die and come away with the casting.

reheat in protective atmosphere, quench in oil, temper for 2 hours, subject to low temperature for 2 hours and then retemper for 1 hour. It was found that a low temperature treatment was indispensable irrespective of the method of carburising or hardening (gas carburising, salt carburising, martempering, etc.) if maximum transformation was to be affected.

In practice heat treatment was carried out in a standard furnace using a cracked dipentene atmosphere. The parts were carburised for 1 hour

TABLE I.—HARDNESS TEST DATA.

Treatment	Hardness Rockwell-C	Hardness Rockwell-15N	Rockwell-C Converted from 15N
As Quenched	59	83	45
Quenched and tempered at 150°C	56	83	45
Quenched and treated at -85°C	64	91	62
Quenched, treated at -85°C and tempered at 150°C	62	91.5	63
Quenched, tempered at 150°C and treated at -85°C	60.5	90	60

at 900° C., the dipentene being pumped into the furnace at a rate of 120 to 130 drops per minute for the carburising period and then completely turned off for ½ hour to allow the case to diffuse rather than to absorb more carbon. At the end of the diffusion cycle the

Subzero Treatment in Hardening Alloy Case Hardening Steels

By H. E. Boyer and H. C. Miller

SUBZERO treatment and its value in transforming retained austenite and the subsequent dimensional stability of hardened steel parts has been a subject to which a great deal of metallurgical research has been devoted during the last four years. Up to the present time, however, emphasis has been focussed on the steels of higher carbon content or high alloy types including the various high speed steels and little attention has been given to the carburising steels of either the straight carbon or alloy types. As the problem of dimensional stability is very serious in making and holding extremely close tolerances on parts of the martensitic types stainless steel and as such tolerances are frequently necessary in the manufacture of certain precision parts, investigations on the subzero treatment of and its subsequent effect on many different type of hardened steels have been carried out. Most recent work has been with the higher alloy types of carburising steels and the present investigation is confined to results obtained on

E-3316 steel. This steel, which contains approximately 0.1% carbon, 3.5% nickel and 1.5% chromium, tends to retain austenite at normal room temperature.

In Fig. 1 are shown two different types of parts which require uniform heat treatment with minimum distortion and maximum stability. Any retention of austenite is very detrimental in the final lapping of the valve hole because heat generated in lapping makes it difficult to control size, and any material which is unstable at room temperature or at operation temperature which is somewhat higher will cause the valve to operate improperly. Standard practice in the heat treatment of such parts has been to pack carburise, slow cool to room temperature,

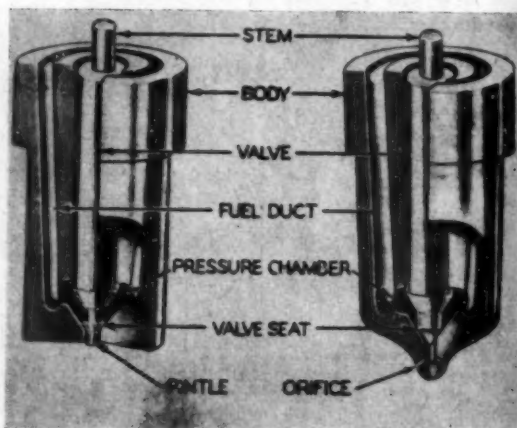


Fig. 1.—On parts of this type Stabilisation is important during both manufacture and use.

dipentene was turned on and allowed to run at 60 to 70 drops per minute to maintain a protective atmosphere against scaling and the part cooled to 785° C. at which temperature it was allowed to remain for 1 hour before quenching directly into oil at room temperature. After quenching the

From *Material and Methods*, 1946, Vol. 23, No. 3, pp. 730-734.

structure showed only relatively few martensitic needles in a matrix of austenite. Hardness test data is given in Table I.

The Rockwell C hardness, after tempering the quenched material for 1 hour at 150° C., dropped to 56 but the superficial hardness remained the same. The microstructure was essentially unchanged. Low temperature treatment at -85° C. for 3 hours brought about a marked transformation in the microstructure, an increase in the Rockwell C hardness and an increase in the superficial hardness. The microstructure after tempering for 1 hour at 150° following the low temperature treatment showed a still more complex transformation,

particularly at the edge of the carburised case as was indicated by the superficial hardness of 15N-91.5. Tempering at 150° before the low temperature treatment at -85° C., produced a noticeable change in the microstructure from that obtained by the low temperature treatment, although it was not as completely transformed as in the case where the subzero treatment was used prior to the tempering operation, and this was substantiated by the hardness reading obtained. The maximum benefit can be obtained by subzero treatment only if used immediately after quenching and prior to tempering in the cycle of operations.

usually 4-10 volts, and a sufficient number of cells are connected in parallel with these to supply the amount of welding current needed. The size of the battery is chosen to have a reserve sufficient to make approximately 2,000 welds in the event of a power supply failure.

The contactor-controller is air-operated and comprises essentially three circular copper alloy plates with connection terminals which plates support four carbon graphite contact discs. The copper mounting plates are water-cooled. The typical contactor of this type is capable of making and breaking welding currents up to 40,000 amp. continuously at 200 times per minute. There is no arcing and no erosion of the graphite contact surfaces.

A copper-covered cast-iron rheostat in the welding circuit is used to control the welding current usually from 50 to 100% of maximum current. The mechanical operation of storage-battery-powered welders is similar to that of conventional a.c. welders. Three-phase full-wave dry-disk-type rectifiers and specially designed controls are employed to convert the usual factory alternating current power to direct current to maintain the storage cells at the proper charge.

Resistance Welding with Storage Battery Power

By J. D. Gordon

STORED - ENERGY welding machines have been developed, based either on the use of electromagnetic induction or the employment of condensers. While these stored-energy machines require about one-fifth of the power of A.C. welders, their cost is high and they have several other disadvantages.

The obvious solution of using accumulators as a means of storing energy has hitherto been prevented by the difficulty of interrupting the enormous low-voltage direct currents. However, the development for this purpose of the carbon-pile rheostat has solved this problem. The use of storage batteries to supply power to resistance welders frees the process from its dependence on high capacity power lines.

The welding capacity of storage battery welders can be expanded merely by increasing the number of cells and the welding rate depends primarily upon the output capacity of the battery charger employed. The range of materials that have been successfully spot- and projection-welded extends from very thin-gauge mild steel, and stainless steel, to heavy-gauge ($\frac{3}{8}$ in.) aluminium alloy sections. Although the full range of possibilities of flash-welding with battery power has not been determined as yet, exceptionally good flash-welds have been made on aluminium and other metals having a narrow plastic range, materials heretofore considered unsuitable for flash-welding.

A new type of cell with internal water cooling is used for the welders. Each cell has ample capacity for an average of 7,000 amp. normally delivered during welding. The cells are connected in series as required to provide the proper welding voltage,

Structural Changes in Carbon and Molybdenum Steels During Prolonged Heating at 480° to 590° C.

By G. V. Smith, R. F. Miller and C. O. Tarr

ENGINEERING structures used for prolonged service at elevated temperatures are generally designed on the basis of creep or stress-rupture data, which are generally obtained in tests of short duration relative to service life. These properties depend on the microstructure of the metal, and inasmuch as metals undergo changes in microstructure when held at elevated temperatures, the creep and stress-rupture tests vary considerably, generally decreasing, throughout the service life. It was therefore considered desirable to study the microstructural changes in steel, occurring over longer periods at the service temperature, or as simulated, though not always equivalent by shorter tests at higher temperatures.

Three bars of S.A.E. 1015 steel containing 0.14% carbon from three heats of similar composition but with different deoxidation practice, six bars of steel containing 0.11 to 0.21%

carbon, 0.48% molybdenum from four heats of similar composition but varying deoxidation practice, and two molybdenum steels of similar composition, one deoxidised with ferro-silicon-zirconium in the ladle and the other with a high aluminium addition in the mould, were selected for examination. The carbon steels were normalised and annealed at 870° C., and the molybdenum steels at 900° C. before heat treatment. Heat treatment was carried out at 480°, 540° and 590° C. for periods of 5, 15, 50, 100, 300, 500, 1,000, 3,000 and 5,000 hours, a separate specimen being used for each period. A Vickers hardness determination and a micro-examination were made on each specimen to determine the results of heat treatment. The progress of spheroidisation and of graphitisation for the various steels after the different periods at the three temperatures are given in Table I, together with their deoxidisation practice.

The results obtained showed that

From *Mech. Eng.*, 1946, 68, 797-801.

From *Proc. Amer. Soc. Testing Mats.*, 1945, Vol. 45, pp. 486-503.

TABLE I. MAIN DEOXIDISERS AND PROGRESS OF SPHEROIDISATION AND OF GRAPHITISATION, AS OBSERVED AFTER 5, 15, 50, 100, 500, 500, 1,000, 3,000 AND 5,000 HOURS AT DIFFERENT TEMPERATURES.

Spec.	Main Deoxidisers		Time to Reach Stage of Spheroidisation, Hours.									Time to Reach Stage of Graphitisation, Hours. (Blank spaces indicate none detected within 5,000 hrs. at temperature.)								
	Ladel lb. per tin	Mould lb. Al per ton	480° C.			540° C.			590° C.			480° C.			540° C.			590° C.		
			Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3
NORMALISED SPECIMENS. MOLYBDENUM STEEL.																				
1	12.6 Fe Si	1.0	100	3000	*	5	1000	3000	5	15	100									
2	12.6 Fe Si	0.0	3000	*	*	5	500	3000	5	50	300									
3	7.7 Fe Si	0.0	5000	*	*	100	3000	5000	50	300	500									
4	7.7 Fe Si	1.5	5000	*	*	50	500	1000	5	50	300									
5	5.1 Fe Si Zr ..	0.0	3000	*	*	500	1000	3000	50	100	500									
6	0.0 Fe Si	3.0	5000	*	*	1000	3000	5000	100	300	1000				5000	*	*	3000	*	*
CARBON STEEL.																				
7	10 Fe Si	1.0	1000	3000	5000	50	500	1000							5000	*	*			
8	10 Fe Si	0.5	300	1000	3000	100	500	1000							5000	*	*	1000	*	*
9	10 Fe Si	0.0	50	500	1000	5	500	1000							5000	*	*	5000	*	*
ANNEALED SPECIMENS. MOLYBDENUM STEEL.																				
1	12.6 Fe Si	1.0	5000	*	*	300	1000	3000	15	100	300	5000	*	*	1000	3000	5000	300	1000	5000
2	12.6 Fe Si	0.0	5000	*	*	500	3000	5000	15	100	500									
3	7.7 Fe Si	0.0	5000	*	*	300	3000	5000	100	300	1000									
4	7.7 Fe Si	1.5	5000	*	*	50	300	5000	5	100	500				3000	*	*	1000	3000	*
5	5.1 Fe Si Zr ..	0.0	5000	*	*	300	3000	*	50	300	500				5000	*	*	1000	*	*
6	0.0 Fe Si	3.0	5000	*	*	500	*	*	100	300	1000				5000	*	*	1000	*	*
CARBON STEEL.																				
7	10 Fe Si	1.0	5000	*	*	50	300	1000	15	100	500				5000	*	*			
8	10 Fe Si	0.5	5000	*	*	50	300	500	15	100	500				5000	*	*	3000	*	*
9	10 Fe Si	0.0	300	*	*	50	300	1000	15	50	300				3000	5000	*		3000	*

* Greater than 5,000 hours (limit of observation).

the hardness of most of the carbon steels increased slightly with time at temperature, then decreased slowly at about the same rate at each of the three temperatures, and the changes appeared to be uninfluenced by the deoxidisation practices used. The increase in hardness was greatest in the annealed (furnace-cooled) steels held at 540° C. The hardness of the annealed molybdenum steels were somewhat similar to those of the carbon steels, but the normalised (air-cooled) steels behaved in a way characteristic of precipitation-hardening systems—that is, the hardness

increased initially, more slowly but to a higher value the lower the temperature, and then decreased.

The rate of spheroidisation of the normalised steels was generally greater than for the annealed steels, as was to be expected from the finer initial structure, but the effect was less marked in the carbon steels. In the normalised carbon steels the rate of spheroidisation was highest in the high titanium-treated steel. In the molybdenum steels the rate increased with increasing silicon plus aluminium addition, whereas a large aluminium addition without silicon was associated

with a lower rate which was probably due to a coarser initial microstructure.

Graphite formed in all the carbon steels, and at about the same rate in normalised and annealed specimens. In the six molybdenum steels, it formed only in the three to which aluminium had been added, and in these three the rate seemed to be faster the higher the silicon content, and faster in the annealed than in the normalised specimens, only one of which showed graphite. In both groups graphitisation was generally faster the higher the temperature within the range studied.



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